

# News Briefs

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## General Developments

*Inquiries about News Briefs, where no contact person is identified, should be referred to the Managing Editor, Journal of Research, National Institute of Standards and Technology, Building 820, Room 126, Gaithersburg, MD 20899-0001; telephone: 301/975-3572.*

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### CRITICAL WAVEFORM MEASUREMENTS SUPPORT NEW OSCILLOSCOPE CALIBRATORS

The unique measurement capabilities of the time-domain waveform measurement systems at NIST were used recently by two U.S. instrument companies to support new oscilloscope calibration products, which these companies have developed. A major company that once dominated this market no longer sells oscilloscope calibrators. Seizing this opportunity, two smaller U.S. firms have introduced calibrators to support the new high-performance digital oscilloscopes that are now available having signal bandwidths as high as 600 MHz. Among other resources, both of the new calibrators provide pulse waveforms with fast, well-defined transitions, which can be used to measure dynamic oscilloscope response characteristics, including transition duration and settling performance. No other national laboratory offers waveform measurements with the requisite uncertainties needed to meet these manufacturers' demanding requirements.

In the most recent tests for one of these companies, NIST was asked to provide characterizations of the pulse waveforms at four amplitudes ranging from 0.25 V to 2.5 V, over a 31 ns time epoch, with an effective 10 ps sampling interval. The waveform measurement data (provided on diskette) are being used by the company to correct the response of the sampling oscilloscope used in the production testing of calibrators. The (corrected) oscilloscope measurements are in turn used to generate numerical corrections for calibrator.

The pulse waveforms of the calibrator are not inherently flat enough to meet specifications without these NIST-traceable corrections. Reported relative expanded uncertainties (95 % confidence) for the waveform measurements ranged from 1.9 % for  $0.3 \leq t < 0.4$ , to 0.4 % for  $2.0 \leq t < 5.0$ , to 0.06 % for  $15 \leq t$ , where  $t$  is the duration in nanoseconds from the halfway point of the waveform transition. Two electrical waveform measurement systems at NIST were used to make these measurements.

### NIST SERVICE SUPPORTS SALES OF U.S. COMMERCIAL FUSED-SILICA CAPACITANCE STANDARD

With a U.S. company claiming that it offers "the world's most stable capacitance standards" in commercial production, NIST is now providing a Special Test, as part of its routine calibration services, for calibrating the company's fused-silica dielectric capacitance standards. Customers can purchase the standards and submit them to NIST to verify the company's data. Better-defined capacitance standards are needed by industry to support the development of advanced electronic instrumentation. Although the best relative uncertainty routinely offered by NIST for gas dielectric capacitance standards is  $(4 \times 10^{-6})$ , careful comparisons made against the reference bank of NIST-built fused-silica capacitance standards (traceable to the NIST Calculable Capacitor—a realization of the SI-defined Farad) have permitted a relative expanded uncertainty of  $(2.4 \times 10^{-6})$  to be assigned to this Special Test service (coverage factor  $k = 2$ ).

Recent efforts by NIST scientists to calibrate the reference bank of NIST-built fused-silica capacitors, which are maintained in a temperature-controlled oil bath, have created a valuable new database history on these standards. This chronological set of measurements provides a reference value that is statistically predicted rather than depending on a value determined at a single time, such as the most recent measured value.

Such a statistical process control has determined that the fractional drift rate of the NIST reference standard capacitors are 15 less than  $(2.5 \times 10^{-8})$  per year, with an average rate of less than  $(3 \times 10^{-9})$  per year, making possible a week's comparison testing using the NIST Type-2 bridge at the  $(5 \times 10^{-8})$  level for the Type A relative standard uncertainty at 1 kHz.

Thus far NIST has provided nine Special Tests on 17 capacitors, provided to eight customers since this service was first started, seven of these tests being completed in this calendar year. The typical turn-around time for a calibration is 3 weeks.

### **BLIND TIP RECONSTRUCTION INCORPORATED IN COMMERCIAL SPM**

A private company has incorporated a version of blind tip estimation into one of its commercial instruments. This technology was developed at NIST and published in a 1994 paper, "Morphological estimation of tip geometry for scanned probe microscopy." It provides a means of estimating tip geometry for scanned probe microscopes by using artifacts to characterize imaging tips. Unique to this technique (and the reason for the word "blind" in its name), the geometry of the artifact need not be independently known. The private company's implementation is used to qualify tips. The tip shape is compared to nominal values and classified as good, worn, or bad, so the instrument user can determine whether the tip needs replacing.

### **TECHNOLOGY TRANSFER CENTER INTERESTED IN IMAGE DEBLURRING TECHNIQUE DEVELOPED BY NIST**

The Mid-Atlantic Technology Transfer Center, College Station, Texas, has contacted NIST with a request for more details on a patent recently given to a NIST mathematician for digital image restoration. The center wants to publicize the invention in their widely disseminated newsletter.

Most image deblurring procedures impose smoothness assumptions on the unknown image in order to stabilize the ill-posed deblurring problem. However, in many important industrial, military, surveillance, or biomedical applications, the desired image is highly nonsmooth, and reconstruction of fine detail is of prime interest. The use of smoothness constraints, required by the mathematician's earlier methods, is particularly ill-advised in medical image deblurring, as this might result in an oversmoothed image in which vital diagnostic information regarding tumors, microcalcifications, and the like, have been eliminated.

The new procedure is based on a new "slow evolution" constraint, which effectively constrains the known blurring kernel, rather than the unknown solution. Since no smoothness constraints are imposed on the image, the procedure is highly effective in reconstructing fine detail. A rigorous analysis of this method appears in the SIAM Journal on Mathematical Analysis, Volume 28, Number 3, May 1997, pp. 656-668. Images deblurred via this procedure have been shown to have higher resolution than those obtained using nonlinear probabilistic approaches such as the maximum entropy method, the Lucy-Richardson method, or the maximum likelihood E-M algorithm, while using far less computer time.

### **ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATION**

During the month of May, the NIST Office of Technology Innovation in Technology Services recommended one innovative technology for commercialization to its Department of Energy partner under the Energy-Related Inventions Program.

Dehumidification Enhancement of Air Conditioners by Desiccant Moisture Exchange—a patented desiccant-assisted cooling cycle consisting of a rotating porous wheel containing a desiccant (moisture absorbing) material installed in a conventional (vapor cycle) air conditioning apparatus. The desiccant material is used in a unique manner to assist the air conditioner cooling coil to dehumidify air.

### **NIST EXPANDS NOISE-TEMPERATURE MEASUREMENT SERVICES TO SUPPORT WR-28 WAVEGUIDE BAND**

NIST scientists have completed construction and verification of a radiometer system for the measurement of the noise temperature of sources having waveguide flanges over a new band, WR-28, 26.5 GHz to 40 GHz. Noise measurements are important in communications, since the presence of noise limits the amount of information that may be transmitted through a given channel. The system offers continuous frequency coverage of the complete band, that includes satellite communications among other applications, and provides the basis for a new measurement service responding to industry needs for measurements of the noise temperature of WR-28 sources. The system constitutes a total-power radiometer, with an internal six-port reflectometer to measure relevant reflection coefficients and a NIST developed liquid-nitrogen cooled reference source. System design is similar to that of existing NIST waveguide systems covering the WR-62 (12.4 GHz to 18 GHz) and WR-42 (18 GHz to 26.5 GHz) bands. It is

capable of measuring sources with noise temperatures from about 50 K to 15 000 K, with typical relative expanded uncertainties of 1.5 % or less (coverage factor  $k = 2$ ) for sources having a noise temperature of about 5000 K and reflection coefficients of less than about 0.1. Two sources already have been received for calibration.

#### **CAPABILITY FOR FABRICATION, TESTING OF VANADIUM DIOXIDE THIN FILMS DEVELOPED TO INVESTIGATE POTENTIAL FOR PHASE-TRANSITION DEVICES**

Three scientists recently developed a local fabrication and testing capability for thin films of vanadium dioxide, a material that is drawing increasing commercial interest. Vanadium dioxide thin films already have demonstrated unsurpassed performance as uncooled bolometers and as optical switches. They form the heart of a number of uncooled infrared imaging systems, which are currently being commercialized by several large manufacturers for surveillance and remote-sensing applications. Most of these devices use relatively amorphous films in the purely semiconducting state; they do not exploit the phase transition at 65 °C. The NIST scientists believe that by exploiting the sharpness in crystalline material of the phase transition with electrothermal feedback, higher performance, in particular speed increases on the order of a factor of 100, will be attainable in current applications and that new applications (e.g., wireless telecommunications at long wavelengths) will become feasible. A NIST interest is the development of bolometers for antenna metrology at quasi-optical frequencies.

The NIST scientists fabricate their films by reactive sputtering of vanadium in an oxygen environment onto heated sapphire substrates. x-ray diffraction measurements indicate that the films' stoichiometry and crystallinity is strongly dominated by the desired vanadium dioxide phase and that the films are highly aligned. Most importantly, resistance versus temperature measurements display the desired metal-semiconductor phase transition at 65 °C. Successful growth of the crystalline material is challenging, but the team has been able to refine its process to achieve phase transitions exhibiting a resistivity contrast of 10 000 to 1. For testing specimen films, the team built a thermal stage using a thermoelectric heat pump to provide a peak-to-peak stability of 3 mK over the temperature range -30 °C to 110 °C.

#### **REMOTE ACCESS AND CONTROL OF SENSORS AND ACTUATORS VIA WWW DEMONSTRATED**

At a recent Sensors Expo held in Boston, two researchers from NIST demonstrated how the IEEE P1451.2 Draft Smart Transducer Interface Standard for Sensors and Actuators can be implemented to access data from remotely located sensors using World Wide Web technology. In that demonstration, a temperature sensor and an actuator were connected to a control network located at NIST in Gaithersburg. From Boston, the temperature sensor was scanned and the cooling fan at NIST remotely controlled via the Internet by means of a modem and commercial telephone service. Live audio and video provided feedback for real-time confirmation of remote command execution.

This demonstration clearly illustrated not only the benefit to be derived from standardization of network interfaces that allow "plug and play" sensors but also the power of Web technology for remote access and control of sensors and actuators.

#### **NIST DELIVERS GROUND-BASED VIDEO DATA TO DARPA**

The goal of NIST's Ground Video Data Collection project is to collect and distribute video and related information that supports computer vision research in applications such as surveillance and monitoring, tactical reconnaissance, and physical security. The project is funded by the Defense Advanced Research Projects Agency (DARPA). The availability of the first set of data has been announced to the DARPA research community. These data consist of a sequence of calibrated video images collected from a camera mounted on the NIST High Mobility Multipurpose Wheeled Vehicle traveling off- and on-road at speeds of 8 km/h to 16 km/h. Most previous video data collections to support research have consisted simply of recorded video. The NIST data collection effort, on the other hand, involves capturing and synchronizing other data with the video. Currently, these data consist of the vehicle's forward velocity and turning rate, collected using an inertial navigation system on-board the vehicle. This inertial data is associated with each image frame and can be used to recover the vehicle's position and orientation when each frame was taken. In addition, camera calibration parameters have been obtained using a large building as a calibration target.

Software for reading the data files and interpreting the inertial data also was made available. The video, inertial data, and camera calibration data, plus supporting software and documentation, are available on-line at <http://isd.cme.nist.gov/staff/coombs/projects/ground-video/>.

### NANOSCALE COULOMB EXPLOSIONS

Using NIST's supercomputers, researchers at NIST and the University of Florida have carried out the first full-scale molecular dynamics simulation of the so-called "Coulomb explosion" phenomenon. This process has been hypothesized to occur when highly charged ions (HCIs) bombard surfaces. The simulations demonstrated the details of mechanisms that can lead to permanent structures on semiconductor (silicon) surfaces at the nanoscale level. The results appeared in the January 1997 issue of *Physical Review B*, and a follow-up study will be featured on the cover of a forthcoming issue of the *Journal of Computational Science*.

The NIST electron beam ion trap (EBIT) team is now busy preparing a complementary set of experiments for silicon surfaces. These will utilize a new scanning-probe microscope that is being installed at EBIT. In an earlier study on an aluminosilicate surface (mica), NIST researchers showed that single HCIs produce feature sizes that can be adjusted by varying the charge of the HCI. On mica, each ion of  $\text{Xe}^{50+}$  produced a feature that appeared to be a cone-shaped protrusion of approximately  $150 \text{ nm}^3$  in volume—huge and remarkably regular on the atomic scale, yet small enough to be of interest for possible applications in nanotechnology.

### HEAVY METALS IN CHEMICALLY PECULIAR STARS

The relative abundances of elements in most stars are similar to that is found on Earth and in the universe as a whole. However, some stars, known as chemically peculiar stars, contain extra proportions of heavy elements. One of the most-studied stars of this type is chi Lupi, which contains 100 000 times more mercury than normal. Stranger still is the fact that this mercury is in the form of the isotope  $^{204}\text{Hg}$ , which comprises only 7 % of natural Hg. Similar anomalies are found in chi Lupi for other heavy elements.

The Hubble Space Telescope (HST) provides a wealth of information about elemental abundances in such stars. Late last summer, NASA researchers, using the Goddard High Resolution Spectrograph (GHRS) on HST, tentatively identified a spectral line from chi Lupi at 155.3 nm as a transition in doubly ionized lead. However, the existing reference wavelength for this line had

been measured with an uncertainty of only 0.01 nm. This was too coarse for GHRS, which can measure wavelengths to 0.0001 nm. Researchers at NIST, using the NIST 10.7 m normal-incidence spectrograph and a hollow cathode lamp containing natural lead, measured the wavelength of the line of interest with an uncertainty of 0.00008 nm. This confirmed the identification of the stellar line as due to lead.

However, a slight difference between the NIST and HST wavelengths suggested that the isotopic abundance of Pb on chi Lupi might not be the natural abundance (52 % as  $^{208}\text{Pb}$ ). The NIST scientists, using only a few milligrams of the pure isotopes  $^{204}\text{Pb}$  and  $^{207}\text{Pb}$  in their apparatus, then measured the difference in their wavelengths. The results showed that  $^{207}\text{Pb}$  (22 % of terrestrial lead) is the dominant isotope of Pb in chi Lupi, confirming an earlier study from Sweden.

### NEW INSTRUMENT FOR CALIBRATING OPTICAL DENSITY SRMS (STEP TABLETS)

Measurements of optical transmission density are important for the medical, photographic, and graphic arts industries as well as for nondestructive testing of a variety of materials. Hundreds of Standard Reference Material units for transmission density, in the form of both x-ray and photographic step tablet films, were available and sold from NIST until a few years ago.

NIST recently has completed development of a new instrument for measuring transmission densities of step tablets, using the diffuse influx method. It is designed to comply with the industrially important ANSI/ISO standards for transmission density measurement. The basis of the instrument is a temperature-controlled silicon photodiode with amplifier electronics capable of measuring signals spanning seven orders of magnitude. This will allow transmission densities as high as 6 to be determined. The instrument is designed to measure automatically many tablets using computerized data acquisition and control of all the relevant components involved in the measurement. This will allow us to measure many tablets reliably, quickly, and routinely.

Procurement of tablets suitable for Standard Reference Materials is under way, and are available for purchase. Customers have been advised through a mailing and in the spring 1997 issue of *Optical Radiation News*.

### NEW STANDARD FOR IODINE-125 BRACHYTHERAPY SOURCES

Very small, sealed "seeds" incorporating the radioisotope  $^{125}\text{I}$  are used in a medical procedure called brachytherapy (from the Greek brachy, meaning near). Arrays of these seeds are arranged in plaque applicators

for contact treatment of eye lesions, or implanted directly into tumor sites for cancer therapy. A report published in early 1996 by the Memorial Sloan-Kettering Cancer Center showed that radiation-seed treatment was a particularly promising approach for treating early-stage prostate cancer. This has led to a rapid growth in the use of these seeds. The major manufacturer in the United States is doubling the size of its plant to keep up with demand, and other companies are entering the market.

NIST hosted a meeting on April 4, 1997, organized by the Council on Ionizing Radiation Measurements and Standards, on progress in the development of new dosimetry standards and measurements for these applications. The meeting brought together representatives from the American Association of Physicists in Medicine (AAPM), seed manufacturers, and NIST scientists to discuss issues in the implementation of new standards and calibrations planned for introduction this winter.

NIST has developed a new standard, based on a new instrument specifically designed to measure the air ionization (air kerma) produced by the radiation emitted from these seeds. This improves upon the old standard and provide calibrations more consistent with the intended use of the seeds. NIST is collaborating with the AAPM and seed manufacturers to introduce the new calibrations into clinical therapy planning so as to deliver the proper dose in conformance with customary treatment protocols.

### **WATER BINDING INTERACTIONS IN PROTEIN CRYSTAL GROWTH**

Protein and DNA crystals play a crucial role in biotechnology, providing atomic-resolution structural information through x-ray diffraction. The database of known structures has grown exponentially in recent years, yet many new structures are needed to facilitate industry goals in metabolic engineering, drug design, and bioinformatics. This increasing demand for diffraction quality crystals is frustrated by the largely empirical trial-and-error methods currently used to determine crystal growth conditions for each macromolecule of interest.

Hydrogen-bonding and water interactions are important in the growth of these crystals as they are in the structure of the macromolecules themselves; each new structure reveals, in addition to the macromolecule of interest, a pattern of bound water molecules in crystal contacts. NIST scientists have analyzed the bound water in the lattice-forming contacts of three polymorphs of the detergent enzyme subtilisin BPN'. By comparing the three high-resolution structures, it was possible to

determine whether the sites occupied by bridging waters sites are similarly occupied in the absence of the crystal contact. It was found that the bridging waters are usually retained in the absence of the contact. This implies that bridging water sites form by overlap of bound water sites in a form of condensation reaction. This further implies that the pattern of bound water on the surface of the protein in solution can determine the geometry of crystal contacts, and thus the formation of the crystal lattice. This work will appear soon in *Protein and Peptide Letters*; meanwhile the investigators have begun to explore systematic changes in crystal growth due to modulation in solvent conditions, e.g., ionic strength.

### **NOVEL ALDEHYDE-TERMINATED SAMs**

Self-assembled monolayers (SAMs) hold great promise in the development of novel biological sensors. A challenging aspect of SAM-based biosensor design is the development of appropriate immobilization chemistries for the attachment of biologically relevant molecules to a surface. A collaboration between scientists at the University of California, Los Angeles and NIST has resulted in the formation and characterization of a novel functionalized SAM that is capable of immobilizing amine-containing molecules to a surface. The new SAM is an aldehyde-terminated alkanethiol monolayer, which is formed by exposing a bare metal surface to an ethanolic solution of a newly synthesized heterocyclic compound, hydroxy-pentamethylene sulfide. Amine-containing molecules in solution react chemically with the aldehyde functionality of the monolayer, resulting in immobilization of the amine-containing molecule on the surface. The aldehyde-terminated SAMs have been characterized thoroughly before and after derivatization with the amine-containing compound by x-ray photoelectron spectroscopy, grazing angle Fourier transform infrared spectroscopy, ellipsometry, and contact angle goniometry. Reaction of the aldehyde surface with amine-containing molecules (in this case, alkyl amines) results in nearly complete derivatization of the aldehyde groups. Future studies will focus on the development of model biosensors formed by derivatization of the aldehyde-terminated monolayer with amine-containing biological molecules, such as proteins and DNA.

### **NIST DETERMINES SULFUR CONTENT OF AEROSPACE SUPERALLOYS AT THE LIMIT OF QUANTITATION**

The U.S. aerospace industry is the nation's leading net exporter of manufactured goods, with jet turbine engines and parts accounting for \$6.1 billion of \$40 billion

in revenue in 1993. The most critical components of all jet engines are the so-called “hot parts,” with the high-pressure (HP) turbine being the most critical. The inlet temperature to the HP turbine sets the upper limit on the power and efficiency of a turbine-based power source. Currently, turbine blades are cast using high temperature (HT) superalloys. These alloys are nonferrous metals that are high in nickel, with moderate amounts of chromium, cobalt, and other refractory elements. The turbine blades are hollow, permitting forced air cooling, while some also are coated with a ceramic thermal barrier. These features permit the operating temperature in the HP turbine to be close to the melting point of the alloy.

Metallurgists believe that the difference between a sulfur mass fraction of 0.5  $\mu\text{g/g}$  and  $\text{mg/g}$  in a superalloy represents the difference between acceptable and unacceptable turbine material. Specifically, the protective oxide coating on the surface of the turbine blades may fail if the mass fraction of the sulfur of the metal substrate is  $>1 \mu\text{g S/g}$ . The instrumental techniques used in the steel and metals industries are almost exclusively combustion based. Accuracy is dependent on calibration using external standards. The aircraft industry and their supporting foundries have asked NIST to measure the sulfur content of some of their new and most technologically advanced superalloys and to certify one or more low sulfur superalloys as an SRM.

The determination of sulfur as the arsenic sulfide molecular ion by isotope dilution thermal ionization mass spectrometry is currently the only method that possesses the required sensitivity and accuracy to address this problem. Below 5  $\mu\text{g/g}$ , the chemical blank becomes the dominant source of uncertainty and the ultimate limitation to accuracy. The blank problem is being minimized by using a least squares approach to the experimental design which allows validation of the blank in relation to its associated samples. A blank and three samples of different mass are processed together and the resultant data linearly regressed. The residual standard deviation using this approach is about 0.05  $\mu\text{g}$  of sulfur, indicating a blank uncertainty of this amount.

Preliminary measurements on nine different superalloy materials submitted by the foundries have confirmed the efficacy of this approach. Superalloy material as low as 0.5  $\mu\text{g S/g}$  has been determined with expanded uncertainties for sulfur near 0.1  $\mu\text{g}$ . From this pool of materials, a candidate SRM has been identified as suitable for certification. Results of these developments have been presented at the two most recent meetings of the Consortium on Casting of Aerospace Alloys.

## SEMICONDUCTOR MANUFACTURERS NOW CAN INCORPORATE SPIN VALVES WITHOUT SPECIAL FABRICATION TECHNIQUE

Magnetic multilayer “spin valve” materials exhibit a desirable combination of a large electrical resistance change and sensitivity to small applied fields that has made them attractive for development of magnetic field sensors, nonvolatile MRAM computer memory chips and especially for development of read heads for ultra-high density magnetic recording. NIST scientists are measuring the effectiveness of techniques to make these spin valves, which have layers only 2 nm or 3 nm thick, less susceptible to damage at elevated temperatures, so that they can be incorporated into existing semiconductor fabrication techniques which involve annealing steps. A promising way to protect spin valves from the thermal degradation is to apply a surface coating as the top layer. The NIST researchers recently have found that 5 nm thick protective coatings of Ta,  $\text{Ta}_x\text{O}_y$ , and Au allow the spin valve materials to survive half-hour anneals at temperatures up to 325  $^\circ\text{C}$ , which is sufficient for some widely used fabrication techniques. While thinner protective layers would be desirable because they shunt less current from the active magnetic layers, the measurements showed that spin valves protected with thinner Au or Cu showed damage after anneals at only 250  $^\circ\text{C}$ .

## UNUSUAL SOLID STATE PROPERTIES OF CUBANE DISCOVERED

Scientists at NIST and the University of Chicago recently have achieved a great advance in understanding the properties of solid cubane ( $\text{C}_8\text{H}_8$ ), an immensely strained molecule that represents an atomic-scale replica of a cube. First synthesized in 1964 by Eaton and Cole at the University of Chicago, the unique geometry of the molecule imposes an angle of 90 $^\circ$  on the C-C-C bond instead of the 109.5 $^\circ$  normally found in other hydrocarbons. Cubane, therefore, possesses a tremendous amount of strain energy, roughly 6.5 eV per molecule. Despite its exceptional structure, cubane remained an academic curiosity until the 1980's when it was recognized that its high heat of formation and very high density made certain derivatives of cubane the potential to be used as fuels or superexplosives. Remarkably, recent results have shown that certain complex cubane derivatives also may have biomedical applications. One, a cubane molecule substituted with keto, cyano, and amide groups, has exhibited activity against the AIDS virus, and another, phenylcubane, has exhibited moderate anticancer activity. Most recently, researchers at

Chicago have found some cubanes are active against bone marrow cancer while other researchers at Northwestern have suggested that activity of yet other cubanes toward monoamineoxidase might presage a use against Parkinson's disease.

Cubane belongs to the class of solids known as molecular solids in which molecules, rather than individual atoms, make up the underlying lattice. Below their melting points, such solids often transform into a different phase, known as a "plastic" phase where these molecules rotate about one or more axes. The crystal structure of such plastic phases is almost always cubic. The recent NIST/Chicago research has determined the structure of the plastic phase of cubane for the first time and probed the details of cubane dynamics. The results of these x-ray and neutron scattering measurements show that the plastic phase of cubane is not cubic but rhombohedral. The researchers were able to explain this surprising result by successfully modeling the potential energy of solid cubane in both the plastic and low-temperature phases. Future work will focus on tetrahedrally substituted cubanes as well as other cubane derivatives to determine how the intermolecular interactions vary with substitution. It is hoped that this will yield valuable insight into the engineering of future cubane-based compounds for specific applications.

#### **NIST DEVELOPS ADVANCED USER INTERFACE TO SUPERCOMPUTING APPLICATIONS**

Researchers recently released WebSubmit, an advanced Intranet application tool that provides a Web page interface to supercomputing applications. It differs from other Web applications because it allows interaction with a user's data files and directories on the target supercomputer as if the user were logged on. The advantage of a Web-based interface is that it is hardware and software independent; it depends only on whatever Web browser the user has available.

The current implementation of WebSubmit provides an interface to a supercomputer (IBM SP2) running the computer's job scheduler (Load Leveler). It supports general use of the scheduler, utility operations on the computer, and an application specific interface to a computational chemistry package. All of the Web pages are dynamically generated with CGI scripts written in Tcl. The Tcl code is modular, making the addition of new interfaces very easy, and simple to customize for a particular super computer site. Future enhancements will include the addition of the NIST crypto toolkit for security, a generalized module constructor, as well as extension to other computer environments. There has been considerable industry interest in the utility.

The WebSubmit Installation Guide, containing directions for downloading program source, is available at <http://www.boulder.nist.gov/websubmit/guide.html>. You can obtain the WebSubmit Users Guide at <http://www.boulder.nist.gov/websubmit/userman.html>.

#### **CONSUMER PRODUCTS TESTED IN LARGE FIRE RESEARCH FACILITY**

Researchers at NIST cooperated with researchers from the Consumer Products Safety Commission (CPSC) in conducting fire tests on consumer goods and residential fire safety equipment. During the past few months, the CPSC has utilized the Large Fire Research Facility to examine devices intended to make halogen lamps safer, investigate conditions under which toaster oven fires occur, and conduct a variety of residential fire safety equipment tests. Two types of residential fire safety equipment were examined: fire blankets and small fire extinguishers. Both products were tested against cooking oil fires on a stove top.

#### **STANDARDS IN TRADE WORKSHOP FOR ANDEAN PACT COUNTRIES, JUNE 2-13, 1997**

On June 2-13, 1997, NIST sponsored a Standards in Trade Workshop for 24 standards and conformity assessment officials from both the government and private sectors of the Andean Pact countries (Bolivia, Colombia, Ecuador, Peru, and Venezuela). This successful workshop was part of an initiative geared to meet U.S. obligations under Article 11 of the World Trade Organization Agreement on Technical Barriers to Trade to offer technical assistance to developing country members and also part of a program to acquaint other countries in key markets of U.S. technology and standards-related practices. The workshop helped to confirm the role of the NIST Regional Standards Attachè in Argentina and in Mexico as knowledgeable technical resources for Latin American countries. They help to facilitate the flow of information on standards and conformity assessment practices between the United States and the Latin American countries and enhance U.S. trade in the hemisphere.

The workshop familiarized the participants with U.S. technology and practices in standardization, conformity assessment, metrology, and measurement systems. Experts from NIST, the American National Standards Institute (ANSI), and other key government and private sector organizations provided briefings on the similarities and differences among our standards and conformity assessment systems as well as the roles of the private and governmental sectors in each system. Activities included visits to NIST laboratories, the Department of Commerce, the Organization of American

States, ASTM, and the Maryland Department of Agriculture's weights and measures laboratory.

The Andean Pact participants proposed shared projects, such as a follow-up workshop to address product certification, laboratory accreditation, and government regulation; and another to address traceability and the establishment of equivalence of national physical standards. A constant focus was maintained on the importance of continued trust and mutual confidence in order to enhance trade in the Western Hemisphere. The professional relationships established during the workshop are expected to enhance follow-up and future collaboration.

### **TESTING STRATEGIES WORKSHOP PROVIDES HANDS-ON INSTRUCTION FOR USE OF "HELP" TOOLBOX**

NIST recently presented its fourth NIST Testing Strategies workshop at the NIST Boulder Laboratories, sponsored in part by the U.S. Air Force Metrology Calibration (METCAL) Program. This was the first Testing Strategies workshop to be held away from the NIST Gaithersburg facilities and the first to be centered around NIST's High-dimensional Empirical Linear Prediction (HELP) Toolbox. The HELP Toolbox is a graphical software package specifically tailored to the application of optimizing the testing of complex electronic devices and instruments. NIST provided intensive hands-on training to the workshop participants on 10 personal computers hosting the HELP Toolbox software.

Participants were from the Air Force METCAL organization; the U.S. Army Test, Measurement, and Diagnostic Equipment organization; the U.S. Naval Warfare Assessment Center; the Sandia National Laboratories; and a private company. In addition, three NIST scientists attended with the goal of applying the principles of testing strategies to microwave calibrations, including measurements made with automatic network analyzers. The attendee from the private company was interested in applying the NIST Testing Strategies approach to the problem of trimming the company's data converter products for optimum performance over a wide temperature range.

The NIST Testing Strategies approach is based on a simple mathematical model that relates the device response at all candidate test conditions to a set of underlying variables. Once an accurate model has been developed, algebraic operations on the model can be used to select an optimum set of test points that will minimize the test effort and maximize the test confidence. The workshop was designed to provide an understanding of the necessary techniques, including estima-

tion of model parameters, prediction of the response of all candidate test points, and the computation of confidence intervals (uncertainty bounds) for the predicted response. The entire process is implemented through the HELP Toolbox.

### **UNIFIED U.S. METRIC STANDARD PUBLISHED**

The ANSI approved, joint IEEE/ASTM SI 10-1997 standard, entitled "Standard for Use of the International System of Units (SI): The Modern Metric System," has now been published. This single, joint standard replaces ANSI/IEEE Std 268-1992, American National Standard for Metric Practice, and ASTM E380-93, Standard Practice for Use of the International System of Units (SI) (The Modernized Metric System). The new standard is the culmination of a 4-year effort by American Society for Testing and Materials Committee E-43, Metric Practice, and Institute of Electrical and Electronics Engineers Standards Coordinating Committee 14, Quantities, Units, and Letter Symbols, to combine their two separate standards into a single national standard.

As a consequence, for the first time in more than 20 years, the United States has but one standard on metric practice. To achieve this single standard, each committee had to give up some of its long cherished positions, such as the way certain unit names and prefixes should be spelled and what position should be taken regarding the everyday use of the term "weight." The new standard is expected to find wide use among the engineers who participate in the work of ASTM and IEEE, especially those involved in writing applications standards. A NIST staff member played a key role in the four-person writing committee (two from ASTM, two from IEEE) that prepared the joint standard.

### **FIRST ROUND OF AIR SPEED PROFICIENCY TESTING PROGRAM INTERCOMPARISONS COMPLETED**

The first round of air speed laboratory testing under the Air Speed Proficiency Testing Cooperative Research and Development Agreement (CRADA) was completed in January 1997. This program was created by NIST and grew out of requests from industry to resolve measurement discrepancies existing among different calibration facilities. Workshops with industry held at NIST established the need to assess the performance of air speed calibration facilities through round-robin testing resulting in the selection of transfer standards and the desired test conditions which span the range from 0.3 m/s to 15 m/s. The transfer package was calibrated in the NIST airspeed calibration facility and then hand-carried by a scientist to the participating laboratories,



where he supervised the intercomparison measurements. Thirteen laboratories, representing instrument manufacturers, DoD, the electrical power industry, and the chemical process industry, are participating in the CRADA.

Representatives from these laboratories met at NIST on May 20, 1997 to discuss the results. In some cases deviations ranged as high as 30 % relative to NIST values. The results will aid participating laboratories in detecting air speed measurement errors and will provide a basis for quantifying and claiming realistic air speed measurement traceability to NIST. To date, two laboratories have corrected measurement deficiencies identified by this program. A second round of laboratory testing is being planned.

#### **CHEMISTRY/ORIENTATION OF LUBRICANTS ON HARD DISK MAGNETIC MEDIA SUBSTRATES**

Two major corporations are collaborating with NIST in the use of ultrasoft x-ray-absorption spectroscopy (USXAS) at the National Synchrotron Light Source, Brookhaven National Laboratory, to probe the surface and bulk of selected lubricant/magnetic-hard-disk systems to determine the bonding and orientation of the lubricant layers at a molecular level. USXAS is an ideal nondestructive tool for studying the disk lubricant system since it has both elemental and chemical sensitivity, and excellent selectivity in carbon bond type. In addition, the average orientation of chemical bonds may be measured via the polarization anisotropy of the soft x-ray absorption spectra. Current investigations are focusing on lubricant aging and wear (i.e., head disk crashes), lubricant surface chemistry, and hard disk surface composition.

#### **NIST HOSTS WORKSHOP ON MICROMECHANICS MEASUREMENT TECHNOLOGIES FOR FIBER-POLYMER INTERFACES**

NIST hosted the Workshop on Micromechanics Measurement Technologies for Fiber-Polymer Interfaces, jointly sponsored by NIST and the Textile Research Institute of Princeton, NJ, May 28-30, 1997. Fifty researchers from industry, academia, and government laboratories attended the workshop to define the current status of measurement technology, demonstrate new automated instrumentation, and identify the critical needs and opportunities for future research. The importance of the fiber-polymer interface for composite performance has inspired considerable research during the last decade on test method development to assess

interfacial micromechanical properties. Although a number of tests have been developed, they all require many assumptions, and comparisons of test results from different laboratories have yielded little or no agreement.

The most important need identified by the attendees from industry was development of relationships between micromechanical testing and the performance of full-scale composites. Composite manufacturers would like to use databases of micromechanical test data on standard composite systems to design a broad array of composite products and to minimize the expensive full-scale testing currently required. However, to develop such databases, test methods and data must be reliable and standardized. Discussions of prior interlaboratory comparisons of test methods revealed that a contributing factor to variability in results came from differences in the sample preparation method. Thus, a standard method of sample preparation was identified as a key need. This led to a suggestion that micro-Raman spectroscopy be used as a quality control tool. In addition, the testing program will call for standardized sample preparation and testing protocols. An important conclusion agreed upon by both industry and academic researchers is that progress is occurring in both data analysis and measurement technology and that industry may be able to rely on a micro-mechanical test within 10 years if similar progress continues.

#### **NIST SYMPOSIUM ADDRESSES YEAR 2000 COMPUTER ISSUES**

On June 9-10, 1997, NIST hosted the International Symposium on the Year 2000: Mastering the Millennium Rollover. More than 350 people from industry, government, and foreign countries attended the meeting. In her keynote address, U.S. Representative Constance Morella described the problems with computing systems worldwide that will result from the century date change on Jan. 1, 2000, if corrective actions are not taken. A private company attendee spoke about raising the level of awareness if the information technology community is to succeed in averting disaster. Speakers from several government sectors indicated the magnitude of the problem for them. Representatives of Australia, Sweden, and the United Kingdom gave overviews of their country's situations. Other speakers covered various aspects of the problem, including legal consequences, "triage," testing, and procurement, and solutions in progress.

Several features of the conference concentrated on specific issues. An entire track explored air traffic management topics. On the second day, a special meeting of the IEEE-sponsored Working Group on

Year 2000 Test Methods attracted many symposium attendees. Twenty companies exhibited tools that could help to solve various aspects of the Year 2000 problem.

### **CCRL ADDS PROFICIENCY SAMPLE PROGRAMS FOR MASONRY MATERIALS**

NIST's ASTM-sponsored Cement and Concrete Reference Laboratory (CCRL) provides programs for evaluating the quality of laboratory testing of construction materials, including the on-site inspection of laboratories and the distribution of proficiency samples. CCRL has initiated three new proficiency sample programs for masonry materials in consultation with ASTM Committees C12 on Mortars for Unit Masonry and C15 on Manufactured Masonry Units, and the masonry trade associations. These programs are the Masonry Mortar Program based on ASTM C270, the Concrete Masonry Unit Program based on ASTM C140, and the Facing Brick Program based on ASTM C67. These programs were started in response to a requirement for proficiency sample testing in C1093 "Standard Practice for Accreditation of Testing Agencies for Unit Masonry." They are in addition to existing CCRL Proficiency Sample Programs for portland cement, portland cement concrete, blended cement, masonry cement, and pozzolans.

### **NIST ULTRASONIC MEASUREMENTS PAPER RECEIVES AWARD**

The American Society for Nondestructive Testing (ASNT) has awarded two NIST staff members their 1997 Outstanding Paper Award. The paper describes a couplant-free technique for performing accurate sound velocity measurements on the rim of railroad wheels when they are either in the field or at a manufacturing facility. By making a quantitative measurement of the difference in transit time for two polarizations of ultrasonic shear waves propagating through the rim of a wheel, the technique can detect and measure the presence of tensile residual stresses that are capable of producing brittle disintegration of the entire wheel. A first prototype instrument to apply this technique in practical environments is now being assembled at NIST. Its intended use will be to prevent wheels with large tensile residual stresses in their rim regions from appearing on in-service railroad cars.

### **NATIONAL CONFERENCE SPOTLIGHTS DATA SECURITY**

How important is stringent security for an organization's information systems? Just ask the Fortune 500 corporation shut down for three days by a logic bomb activated by a contractor. Or the apparel manufacturer

that had personal data on 20 000 employees stolen from its database.

With the typical high-technology theft resulting in a \$2 million loss, it's no wonder that people worldwide have looked to the annual National Information Systems Security Conference to hear from experts about the hottest security issues. The 20th conference, held at the Baltimore (Md.) Convention Center, Oct. 7-10, 1997, provided a unique international forum for discussing, debating and understanding critical topics such as:

- securing electronic commerce,
- Internet and World Wide Web security,
- protecting trade secrets and company assets,
- preventing computer crime and cellular phone fraud,
- firewall technology,
- virus detection and elimination, and
- encryption and cryptography.

The NISSC is co-sponsored by NIST and the National Security Agency's National Computer Security Center. It is held in parallel with the Information Systems Security Exposition (sponsored by the Armed Forces Communications and Electronics Association), which showcases the latest information systems security technologies.

For more information on NISSC, call (410) 850-0272 or check out the NISSC homepage at <<http://csrc.nist.gov/nissc/>>.

### **UNITED STATES, NEW ZEALAND EQUALIZE ACCREDITATION PROGRAMS**

NIST's National Voluntary Laboratory Accreditation Program and International Accreditation New Zealand recently signed a letter extending an earlier memorandum of understanding that recognized the equivalence of accreditation programs for calibration and testing laboratories in the two countries. Mutual recognition of the two accreditation programs means that test and calibration data from labs accredited by either nation will be accepted by both, greatly facilitating trade.

To reach the current agreement, both nations conducted extensive evaluations of the other's calibration and testing laboratory accreditation programs. They first assessed how well their counterpart's system complied with the requirements of ISO/IEC Guide 58, Calibration and Testing Laboratory Accreditation Systems-General Requirements for Operation and Recognition. Then, each standards body sent representatives to the other nation to conduct on-site reviews of a sampling of laboratories for compliance with ISO/IEC Guide 25, General Requirements for the Competence of Calibration and Testing Laboratories.

For more information, contact James L. Cigler, NVLAP, Room 282, Building 820, NIST, Gaithersburg, MD 20899-0001, (301) 975-4171, <james.cigler@nist.gov>.

### **NEW U.S. SYSTEM FOR LAB ACCREDITATION STARTS MAY 1998**

Designed as an open voluntary system to bring “order out of chaos” in laboratory accreditation, the National Council for Laboratory Accreditation (NACLA) has targeted May 1998 as the date it plans to be fully operational.

Laboratory accreditation is necessary to give confidence to all users of a testing or calibration laboratory’s results. Such confidence assures the public that products purchased are safe, reliable and of good quality. In turn, national and international recognition of a lab’s competence eliminates multiple, often duplicative testing before a product can be sold in different markets.

NACLA, a partnership between the public and private sectors with strong support from NIST, is designed to provide a uniform approach for recognizing accrediting organizations and laboratories as competent to meet international standards. The goal is for a testing or calibration laboratory accredited in a given field by a NACLA-recognized body to have worldwide acceptance of its test data. Therefore, one test of a product in such a laboratory would open it to the entire global marketplace.

An interim board of directors has been elected to develop the operational procedures and bylaws of NACLA. These will be presented for public review by May 1998 at a series of NIST-sponsored workshops. Upon their acceptance by the NACLA membership and the election of a new board of directors, the body will be operational.

For more information on NACLA, contact Belinda Collins, Room 282, Building 820, NIST, Gaithersburg, MD 20899-0001, (301) 975-4000, <belinda.collins@nist.gov>; David Krashes, Mass Materials Research Inc., P.O. Box 810, W. Boylston, MA 01583, (508) 835-6262; or Fred Grunder, American Industrial Hygiene Association, 2700 Prosperity Ave., Suite 250, Fairfax, VA 22031-4320, (703) 849-8888, <fgrunder@aiha.org>.

### **REPORT “SCOPES OUT” DETAILS OF MICROCALORIMETER**

In December 1996, NIST announced the development of a revolutionary new x-ray microcalorimeter that offered more precise materials analysis, especially for the semiconductor industry. The advanced-design spectrometer fits easily onto a commercially available scanning electron microscope and achieves x-ray

resolution that is at least 10 times better than most conventional products. A 25-page technical paper is now available that describes the device in detail.

SEMATECH, the semiconductor industry’s manufacturing research consortium, believes that microcalorimeter detectors “have the potential to become the x-ray detector of choice for particle identification and other semiconductor characterization applications.” NIST recently received a patent for its microcalorimeter (and filed several more) and is looking for industrial partners to share in the instrument’s commercial development.

To obtain a copy of paper no. 22-97, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, <sarabeth@boulder.nist.gov>. For technical information, contact John Martinis, Div. 814.03, NIST, Boulder, CO 80303-3328, (303) 497-3597, <martinis@boulder.nist.gov>.

### **PAPER TRACKS HISTORY OF WELDING SOFTWARE**

A new paper from NIST reviews the worldwide growth and trends in welding software over the past 10 years. It discusses these trends in terms of changes in computer resources in this area. It also identifies the development of sophisticated welding applications for these computer resources.

The first U.S. meeting to address the application of computers to welding was a Knoxville, TN., workshop in 1986 sponsored by NIST and the American Welding Institute. It helped identify welding applications for software developers and produced a ranked list of database needs. Since that time, the workshops have continued on a biannual basis emphasizing software development and its application to welding. This past July saw the workshop join with its European counterpart to become the Seventh International Conference and Exhibition on Computer Technology in Welding.

One trend covered by the paper is the migration from main frames to personal computers. For example, at the 1986 workshop there was a discussion of five welding programs for mainframes and only one for personal computers; by 1994, there were only three programs for mainframes and 35 for personal computers. The paper also discusses the history of welding software in other parts of the world, particularly the United Kingdom and Germany. This latest document complements an earlier report, which lists welding software available for sale.

For a copy of paper 21-97, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, <sarabeth@boulder.nist.gov>. She also is the source for the list of available welding software; ask for paper no. 10-96.

## PROPOSALS SOUGHT FOR PRECISION MEASUREMENT GRANTS

NIST is seeking project proposals for two research grants for fiscal year 1999 in the field of precision measurement and fundamental constants. Each Precision Measurement Grant of \$50,000 for one year may be renewed by NIST for up to two additional years for a total of \$150,000. Prospective candidates must submit summaries of their proposed projects and biographical information to NIST by Feb. 2, 1998.

NIST Precision Measurement Grants are awarded each year to faculty members of U.S. universities or colleges for work in determining values for fundamental constants, investigating related physical phenomena or developing new, fundamental measurement methods. By the Feb. 2, 1998, deadline, applicants should submit a pre-proposal summary of not more than five double-spaced pages outlining the objective, motivation, and technical approach of the research and the amount and source of current funding for the research, together with a concise biographical sketch of the applicant and a list of the applicant's most important publications. On the basis of this material, four to eight semifinalists will be selected by the NIST Precision Measurement Grants Committee and the Outside Review Committee to submit more detailed proposals.

Submit three copies of the pre-proposal summary to Barry N. Taylor, B161 Technology Building, NIST, Gaithersburg, MD 20899-0001. For more information, contact Taylor at (301) 975-4220, or visit the Precision Measurement Grants World Wide Web page at <http://physics.nist.gov/ResOpp/grants/grants.html>.

## FUNDAMENTALLY NEW ENGINEERING TOOL HONORED

A powerful, yet fast and versatile simulation technique for estimating machining errors in the dimensions and geometry of parts has earned an R&D 100 Award for a NIST-based collaboration of university, industry, and government researchers.

Described as a "fundamentally new" engineering tool, Machining Variation Analysis was developed by a team from NIST, the Massachusetts Institute of Technology, and a Pennsylvania maker of grinding equipment. With the innovation, engineers can predict the true shape of a manufactured part, accounting for the influences of a machine tool's random and systematic errors. It also provides machine-tool designers with a means of immediate feedback. For alternate combinations of components, they can evaluate performance tradeoffs and then optimize specifications for building and assembly. The private company engineers used MVA to fine tune the build specifications for its new crankpin grinding machines.

The software tool exploits four powerful mathematical methods. Analyses requiring days of computing with other types of simulation software can be completed within minutes. As a result, manufacturing engineers quickly can estimate machining accuracy and repeatability over production runs of thousands of parts.

For more information, contact Johannes Soons, B102 Sound Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-6474, [soons@nist.gov](mailto:soons@nist.gov). At MIT, contact Daniel Frey, System Design and Management Program, (617) 253-7068, [danfrey@mit.edu](mailto:danfrey@mit.edu).

## ALGORITHMS SOUGHT FOR ADVANCED ENCRYPTION STANDARD

NIST invites security product manufacturers, voluntary standards organizations, research organizations and others to submit candidate algorithms that could become the basis of an Advanced Encryption Standard, according to a notice in the Sept. 12, 1997, issue of the Federal Register.

"It is intended that the AES will specify an unclassified, publicly disclosed encryption algorithm available royalty-free worldwide that is capable of protecting sensitive government information well into the next century," the notice says.

Once the multiyear development effort is complete, the AES will be used by agencies of the Federal Government and will be available for voluntary use by companies, state and local governments, and others who wish to use a strong encryption standard that is backed by the Federal Government.

In addition to describing what makes a complete submission package, the notice lists the minimum acceptability requirements for a candidate algorithm as well as the evaluation criteria that will be used to assess and compare the submissions.

Candidate algorithm submission packages must be received by June 15, 1998. Any submission packages received before April 15, 1998, however, will be reviewed for completeness by NIST, and these early submitters will be notified of any specific deficiencies by May 15, 1998. This will allow time for deficient packages to be amended by the final submission deadline.

The Federal Register notice is available at [http://csrc.nist.gov/encryption/aes/aes\\_9709.htm](http://csrc.nist.gov/encryption/aes/aes_9709.htm). Submission packages should be sent to the director of the Information Technology Laboratory, Attn: Advanced Encryption Standard Nominations, A231 Technology Building, NIST, Gaithersburg, MD 20899-0001. For general information, contact Edward Roback at (301) 975-3696, fax: (301) 948-1233, [aesquest@nist.gov](mailto:aesquest@nist.gov).

### NEW NIST DATABASE AVAILABLE FOR EVALUATING ROCKET FUEL PROPELLANTS

A new database from NIST's Standard Reference Data Program, the NIST Shomate Coefficients Database, provides thermochemical data for the evaluation of performance criteria for fuels and propellants for rockets and jet engines. The database is specifically designed for those who use the propellant evaluation program code from the Naval Weapons Center at China Lake, CA. The program used at China Lake can handle up to 12 chemical elements and 200 combustion products.

Aerospace engineers can make use of the NIST Shomate Coefficients Database to calculate high temperature chemical equilibrium and performance characteristics of propellant systems. The equations provided will reproduce the JANAF (Joint-Army-Navy-Air Force) Thermochemical Tables with an uncertainty less than that of the actual tables. The coefficients are available in the Shomate and the Cruise formats, the latter being that required by the China Lake code.

The NIST Shomate Coefficients Database requires 1.5 MB available hard disk space, an 80386, 80486 or Pentium processor with an operating system of PC-DOS or MS-DOS 3.3 or higher. Standard Reference Database 50 is available from the NIST Standard Reference Data Program, (301) 975-2208, fax (301) 926-0416, e-mail <srdata@enh.nist.gov>, or visit <<http://www.nist.gov/srd>>.

### FQA STATUS: CHANGES PROPOSED, MORE LABS ACCREDITED

The Fastener Quality Act—the national program to protect public health and safety by ensuring that certain nuts, bolts and other fasteners used in critical situations (such as attaching aircraft engines to fuselages) conform to specifications—recently reached two milestones toward its planned implementation in May 1998.

First, NIST is seeking public comment on a proposed major change to the regulations. Announced in the Federal Register of Sept. 8, 1997, the proposed change would allow accreditation of manufacturing facilities that utilize quality assurance systems, such as QS-9000 for statistical control during processing. The regulation previously required that testing and approval for each lot of fasteners be done at the end of the production line and only by an accredited testing laboratory.

The new language addresses the concerns of the automotive industry that on-line quality testing is much more effective than lot sampling and should be recognized under the FQA regulation. Since end-of-line testing is typically invoked to confirm the stability of the in-process statistical control system, an accredited testing laboratory still must conduct that testing.

Other proposed changes to the regulations address: (1) passing laboratory inspection and testing reports along the supply chain to the fastener manufacturer, (2) the issue of removing manufacturer or grade identification markings at the customer's request, (3) "grandfathering" fasteners and (4) revised definitions, clearer text and corrections to editorial errors in certain sections.

NIST's Office of Standards Services, which manages the implementation of the FQA, also reports that by Aug. 31, 1997, nearly 400 U.S. testing laboratories had applied for accreditation in compliance with the act. It is expected that the majority will successfully complete the process, putting the number of accredited labs by May 1998 at the level needed to fully implement the FQA.

NIST urges all testing laboratories that want accreditation but have not begun the process to do so immediately. This will ensure that they achieve accreditation before the FQA implementation date of May 26, 1998.

For information, contact Subhas G. Malghan, Room 306, Building 820, NIST, Gaithersburg, MD 20899-0001, (301) 975-5102, fax: (301) 975-2183, <[subhas.malghan@nist.gov](mailto:subhas.malghan@nist.gov)>, or see the FQA page of NIST's Web site at <<http://www.nist.gov/fqa>>.

### ROOM TEMPERATURE METHANE DETECTOR DEVELOPED

A fast, accurate instrument for spectroscopically measuring the concentration of methane and other gases, such as carbon monoxide, carbon dioxide, and nitrous oxide, in air has been developed by researchers from NIST, Rice University, the National Oceanic and Atmospheric Administration and Stanford University.

The instrument is based on room-temperature solid-state lasers and has a resolution of one part in  $10^9$  in measuring concentrations. The system is simpler, faster (one measurement per minute) and more portable than the chemical processing and gas chromatography methods currently used.

Methane and other organic gases absorb infrared light with wavelengths of 3  $\mu\text{m}$  to 5  $\mu\text{m}$ , far outside the normal emission range of room temperature diode lasers. However, suitable probe light can be generated by combining the light from an 808 nm diode laser with the 1064 nm light from a compact diode-pumped, solid-state neodymium: yttrium-aluminum-garnet (known as Nd: YAG) laser. Combining the beams in a crystal of periodically poled lithium niobate creates a single beam with a wavelength of 3.4  $\mu\text{m}$ . This light can be tuned over a range of wavelengths, so that it can be used to measure and differentiate between methane and water,

and even between methanes containing different carbon isotopes (carbon-12 and carbon-13).

For a technical paper (no. 28-97) describing the instrument, contact Sarabeth Harris, MC 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, <sarabeth@boulder.nist.gov>.

### SOFTWARE IMPROVES PROCESSING OF NEAR-FIELD MEASUREMENTS

Microwave and millimeter-wave antenna measurements are often made by taking many readings (typically  $10^4$  to  $10^6$ ) close to the antenna and then mathematically processing the data to map the antenna's far-field pattern. Normally, accuracy depends on positioning the measurement probe precisely on a rectangular grid of points spaced across the antenna's aperture.

NIST has developed new software for processing planar near-field antenna measurements taken where probe positioning cannot be maintained precisely, such as at high frequencies or in field tests. The software implements a more efficient method for transforming measured data to obtain the antenna's far-field pattern. Other methods designed to handle nonideal, but known, probe positions require time-consuming calculations. However, this method can efficiently remove probe position errors during processing if probe positions are known, for example, from independent laser interferometry measurements.

The software also can process near-field data measured in other scanning geometries such as plane-polar or plane-bipolar. By relaxing the mechanical tolerance requirements of positioning, the error correction capability can extend greatly the frequency range of existing microwave antenna measurement systems that use near-field scanning methods.

For more information regarding the theory behind the software and its availability for evaluation, contact Michael H. Francis (303) 497-5873 <francis@boulder.nist.gov>, Bradley K. Alpert (303) 497-5920 <alpert@boulder.nist.gov>, or Ronald C. Wittmann (303) 497-3326 <wittmann@boulder.nist.gov>. A paper (no. 34-97) describing this work is available from Sarabeth Harris, MC 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, <sarabeth@boulder.nist.gov>.

### NEW IMAGING TECHNIQUE STRONG ON POWER, GENTLE ON SAMPLES

There's a new type of kinder, gentler microscopy. Near-field scanning optical microscopy (abbreviated NSOM) is an emerging technique that combines the nondestructive advantages of optical microscopy with

nanometer-scale resolution near that of atomic force or electron microscopes.

NSOM was developed by NIST physicists working in collaboration with researchers at the University of Virginia and the Naval Research Laboratory. The technique works by channeling laser light through a fiber optic probe, scanning it about 10 nm above a sample surface, and then collecting it on the other side. An opening at the tip of the probe is only about 50 nm in diameter, smaller than a wavelength of visible light but large enough for a small portion of the photons to escape.

The new technique should be useful for measuring nanometer-scale optical properties of waveguides and other fiber optic communications components, as well as for viewing delicate biological samples and for characterizing nanometer-scale structures and defects during semiconductor manufacturing.

Other NIST research groups are working on additional applications for NSOM, including nanometer-scale chemical composition analysis.

For technical information about the NSOM project, contact Lori Goldner, A320 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3792, <lori.goldner@nist.gov>.

### PARTNERS SEEK BETTER TECHNOLOGY THROUGH THE LASER GLASS

NIST and a private company have signed a cooperative research and development agreement to look together at different types of glass used for carrying laser light in photonic systems. They also will try to determine why ultraviolet light causes changes in the index of refraction in optical fibers and bulk glass.

The company will supply novel phosphate glass and silicate rare-earth-doped glass compositions for which NIST will provide waveguide fabrication services and measurement support for waveguide and laser characterization (including optical near-field analysis, mode index measurements, loss measurements, lifetime measurements, spectroscopic measurements and laser performance measurements).

In addition, the two partners will study UV sensitivity of bulk glass samples. The goal is to understand the physical mechanism responsible for UV light-induced index of refraction change in glass. This information could lead to greater stability of and improved production methods for the components in optical fiber and planar waveguides currently manufactured using this phenomenon. For this part of the CRADA project, the company will provide glass samples and glass chemistry expertise to NIST, which will investigate techniques for measuring the samples' UV photosensitivity.

For more information about this research, contact (1) David Veasey, NIST, (303) 497-5952, <veasey@boulder.nist.gov>; (2) Sarah Gilbert, NIST, (303) 497-3120, <sgilbert@boulder.nist.gov>; or (3) Joseph Hayden, Schott, (717) 457-7485, ext. 351.

### **NIST/NSA TEAM TO ENHANCE INFORMATION SECURITY QUALITY**

In a move to boost the international competitiveness of U.S. information security technology producers, NIST and the National Security Agency have established the National Information Assurance Partnership (NIAP). The partnership will employ the latest techniques to develop product specification tools, testing methods and tests so that testing laboratories and organizations—as well as consumers and producers of information technology products—will have objective measures for evaluating quality and security.

Consumers need confidence and assurance in the products and technologies they use to protect valuable information. That confidence is bolstered when the products have been tested and certified by an independent organization. The NIST/NSA effort is designed to foster the creation of such organizations and the certification processes they will use. The NIAP has several goals, including promoting demand and investment in security-enhanced products; moving current evaluation and testing efforts from the federal government to accredited, private-sector laboratories; and fostering research and development in security tests, test methods and metrics.

### **ANTENNA REDUCES AMBIENT FIELD CALIBRATION ERRORS**

The market for wireless communication products has skyrocketed during the past few years. To meet the ever growing consumer demand, manufacturers of these items have had to increase production without sacrificing product quality. Such quality control, in turn, requires ever higher levels of calibration accuracy.

For example, manufacturers must be able to measure accurately the performance characteristics of both their in-house standard antennas and the antennas they design for commercial use. One type of antenna facility commonly used for electromagnetic compatibility testing is affected by ambient electromagnetic fields. These fields degrade the signals being assessed and limit the measurement accuracy.

NIST has developed a standard radiofrequency antenna that improves discrimination of out-of-band interference while maintaining the integrity of the

signal. The key element is a new rf electro-optic modulator that converts the electrical signal from the antenna to a light wave that can be “piped” to the measuring equipment by optical fiber. The nonconducting fiber is immune to electromagnetic interference and does not perturb the electromagnetic field being measured.

For more information, contact Motohisa Kanda, MC 813.07, NIST, Boulder, CO 80303-3328, (303) 497-5320, fax: (303) 497-6665, <mkanda@boulder.nist.gov>.

### **INTERNATIONAL ON-WAFER MILLIMETER-WAVE MEASUREMENT COMPARISON TRANSFERS NIST METHODOLOGY TO HELP INDUSTRY MAKE BETTER MEASUREMENTS**

A NIST researcher has collaborated with a major U.S. electronics company, and l’Institut d’Electronique et de Microlélectronique du Nord (IEMN), a laboratory in Lille, France, to implement an international on-wafer measurement comparison program at millimeter-wave frequencies. Millimeter-wave integrated circuits are becoming increasingly important in wireless communications, automotive electronics, and alternatives to conventional cable television technologies. The study established the accuracy of on-wafer calibration and measurement verification procedures for on-wafer measurement equipment at millimeter-wave frequencies. The comparison helps the electronics company and other participants in the comparison to implement these measurement methods in their laboratories and to verify their measurement accuracy. This work also helps to ensure in a concrete way the recognition and acceptance of the NIST on-wafer calibration and measurement methods not only in the United States but abroad as well.

Early studies predicted that multiple modes would propagate in co-planar waveguide (CPW) at millimeter-wave frequencies, causing the NIST-developed benchmark, the multiline thru-reflect-line (TRL) calibration, to fail. NIST, the electronics company, and IEMN first demonstrated the applicability of this benchmark calibration at millimeter-wave frequencies: the measurements made at the company and IEMN demonstrated convincingly that the TRL algorithm accurately measures a single CPW mode of propagation. The researchers also tested a NIST-developed comparison procedure at millimeter-wave frequencies and assisted the electronics company in implementing and running an international millimeter-wave on-wafer measurement comparison. A number of laboratories in the United States already have benefited from participation in this measurement comparison.

## **NIST TRACEABILITY FOR RADIOASSAY LABORATORIES**

Hundreds of thousands of radionuclide measurements are made each year to support environmental monitoring and cleanup, decontamination and decommissioning efforts, testing for contamination of radiation workers, and health and safety at commercial manufacturing operations. Many of these measurements support governmental requirements, and are monitored by performance evaluation programs, e.g., at EPA or DOE. In most cases, these programs have not been traceable to NIST.

The American National Standards Institute (ANSI) recently released Standard N42.23, "Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories," as guidance to address this serious national quality assurance shortcoming. This standard was written by a committee representing the commercial radioactivity laboratories, nuclear power radiochemistry laboratories, academia, DOE, NRC, EPA, States, TVA, and NIST. It provides guidance for service laboratories to demonstrate traceability to NIST through a chain of unbroken comparisons, using intermediate Reference Laboratories. Furthermore, the standard focuses on criteria for a national accreditation program, based on demonstrated traceability to NIST, and the associated quality assurance and testing protocols. Responsibilities are outlined for the Accrediting Organization, NIST, and the Reference, Monitoring, and Service Laboratories.

The concepts from this standard already are being used by the National Environmental Laboratory Accreditation Conference and the DOE Radiobiassay Laboratory Accreditation Program.

## **NIST DEVELOPS NEW REFRIGERATION SYSTEM DESIGN TOOLS**

With the advent of new zeotropic refrigerant mixtures as replacements for CFCs in the refrigeration industry, completely new equipment/system design tools are required. NIST's plan is to develop the basis for artificial-intelligence-based design tools that automatically optimizes performance. The first step is now complete. NIST researchers have developed a new software package, EVAP-V, for the design of air-to-refrigerant evaporators. It consists of a first principles detailed simulation model that uses NIST's electronic database REFPROP for calculating thermophysical properties, and a Visual C++ interface. The Visual C++ interface was developed in cooperation with researchers from the George Mason University. Used as a preprocessor, the interface allows for graphical specification of complicated evaporator circuitries. After simulation is completed, the interface

displays detailed performance information for each tube in the evaporator circuitry including refrigerant temperature, enthalpy, entropy, quality, and pressure drop. The beta version of EVAP5-V has been provided to 12 equipment manufacturers for testing.

## **ELECTROGALVANIZED ALLOY COATING THICKNESS STANDARDS PROVEN FEASIBLE**

In recent years, electrogalvanized coatings on steel have developed well beyond simple elemental deposits into rather sophisticated alloys such as Zn-Ni, Zn-Fe, and Al-Zn. Today, pure zinc still maintains about 82 % of the domestic market, followed by Zn-Ni and Zn-Fe at about 9 % each. Coating thickness and composition standards, required for the calibration of on-line x-ray fluorescence instruments and gravimetric techniques (weigh-strip-weigh) for process control of continuous strip plating, have failed to keep pace with the development of the alloys. To address this shortcoming, research efforts at NIST have concentrated on obtaining uniform zinc electrodeposits with a nominal thickness between 2  $\mu\text{m}$  and 5  $\mu\text{m}$ . Characteristic electrodeposits for the cell geometry employed have been contour-mapped to identify the region affected primarily by the edge effect and the size and location of areas complying with a 5 % uncertainty in thickness distribution. Results indicate the feasibility of the production of small coupons (3 cm  $\times$  3 cm) intended for standards for laboratories using the gravimetric techniques, and work continues on the optimization of the process to allow production of larger coupons (10 cm  $\times$  15 cm) for the on-line wide beam x-ray fluorescence instrumentation.

## **MELTING STUDIES AT NIST IMPROVE THE TECHNOLOGY OF HIGH-TEMPERATURE SUPERCONDUCTOR WIRE AND TAPE**

Superconductor tape technology has been advanced significantly using high-temperature superconductor materials in the  $\text{Bi}_2\text{O}_3\text{-PbO-SrO-CaO-CuO}$  (Pb-Bi-SCCO) system. Pilot manufacturing of long-length composite tapes based on the 110 K Pb-2223 phase (nominally  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ ) has produced a variety of prototypes, including superconducting motors, magnets, transformers, and transmission cables, all capable of operating at liquid nitrogen temperatures.

Much of the progress has resulted from improved powder-in-tube (PIT) processing techniques, particularly in the area of grain texturing, which is required for producing large critical current densities. Liquid formation during processing promotes superconductor grain growth and alignment, both of which improve electrical performance. Knowledge of the liquid phase involved



in the formation of the superconductor, including its primary crystallization field, is essential for large-scale applications. Phase equilibria and phase diagrams provide the framework for controlled melting of the superconducting phases. NIST has established a team with a comprehensive research facility for elucidating phase equilibria in the Pb-BiSCCO system. This project is partially supported by the Department of Energy, with further collaborations with processing groups at Argonne National laboratory, Los Alamos Laboratory, Oak Ridge National Laboratory, and a private company. Accomplishments include the location of the primary crystallization field of the 80 K high- $T_c$  (critical temperature) 2212 BiSCCO phase, and a preliminary outline of the five-component Pb-2223 crystallization field. These results can be used to improve processing by bracketing the optimum temperature and compositional range to obtain liquid in equilibrium with the superconducting phases.

#### **NIST CALCULATIONS REFUTE INTRINSIC-IMPEDANCE HYPOTHESIS FOR AC QHE MEASUREMENTS**

NIST researchers have completed calculations to test a hypothesis devised to explain a serious anomaly reported in connection with measurements of ac quantized-Hall resistance. Their results indicate that the hypothesis is not valid.

Recent experiments at several national metrology laboratories found that the value of the ac quantized-Hall resistance was apparently frequency dependent and did not extrapolate to the dc quantized-Hall resistance value at zero frequency. It was suggested that these results are due to intrinsic impedances in the quantum-Hall effect devices. The researchers carried out calculations of the internal capacitance, the kinetic inductance, and the magnetic inductance of quantum-Hall effect devices to see if these intrinsic impedances would be expected to affect significantly the value of the ac quantized-Hall resistance. The capacitances and inductances are obtained using the potential, electric field, current, and current density distributions calculated earlier by one of the researchers for quantum-Hall effect resistance standard devices. The integrals were solved analytically, rather than numerically, with no free parameters. The predicted impedances yield only small, out-of phase corrections to the ac quantized-Hall resistance. Thus intrinsic impedance should not be a significant problem in ac quantum-Hall resistance measurements.

There are, however, capacitances, inductances, and resistances associated with external lead connections to

the devices, with electrical shields around the devices, and with contact resistances to the devices. The impedances of these additional circuit elements also must be accounted for if an impedance standard is to have the intrinsic in-phase value of the quantized Hall resistance. A paper describing this work has been published in the NIST Journal of Research.

#### **HETEROLOGOUS EXPRESSION OF SEVEN-TRANSMEMBRANE-HELIX PHOTORECEPTOR**

Rhodopsin, the photoreceptor protein that mediates dim-light vision, is composed of a single 348 amino acid residue polypeptide chain, opsin, and an 11-*cis*-retinal chromophore. The opsin protein folds into a structure of seven membrane-spanning helices, making it a popular model for the large family of seven-transmembrane-helix receptors involved in a wide variety of cellular signal transduction events. This membrane protein family has long been a key interest of research groups in the biotechnology industry because of its importance as targets for therapeutics and drug design. Painstaking efforts to obtain high-resolution structural information for these proteins in general, and for rhodopsin in particular, have failed in part due to the low levels of material, low stability, and inherent light sensitivity. Consequently, industry has turned to biochemical approaches to investigate the structural attributes of this family and to obtain useful variants for more definitive studies.

In an effort to address this need, researchers at the Center for Advanced Research in Biotechnology (CARB) have developed a highly efficient expression system capable of producing post-translationally modified rhodopsin, as well as defined domains, in quantities required for detailed biophysical and structural analyses. This development (NIST, the University of Maryland Biotechnology Institute, and Montgomery County MD, established CARB in 1984.) was achieved by constructing an expression plasmid specific for the methylotropic yeast *Pichia pastoris* that contains the bovine opsin gene. Quantitative-competitive polymerase chain reaction (PCR) analysis of a stable yeast transformant indicated that a single copy of the opsin gene was integrated into the yeast genome. Utilizing this construct, up to 0.3 mg fully functional rhodopsin can be expressed per liter of culture, which represents a considerable quantity of a membrane protein. This advance will allow for the high level of expression of mutants of rhodopsin as well as related seven-transmembrane-helix receptors for more extensive and detailed structure/function studies.

## DEVELOPMENT OF A NEW LOW-PRESSURE STANDARD

The existing national primary standards for low pressures in the United States are the ultrasonic interferometer manometers (UIMs) developed at NIST that use mercury as their manometric fluid. The manometers, which are state of the art in their design and construction, are characterized by an uncertainty due to systematic effects of  $18 \text{ mPa} + 5.2 \times 10^{-6}$  times the pressure and statistical uncertainty of 1 mPa to 3 mPa (coverage factor  $k = 2$ ). However, full benefits of the high precision of the mercury UIMs cannot be realized because the lowest absolute pressure that can be measured is limited by the relatively high vapor pressure of mercury (0.2 Pa at room temperature).

These limitations have been overcome by the recent development of a new low-pressure range UIM standard that uses an oil (di-ethyl hexyl sebacate), whose vapor pressure is below  $10^{-3}$  Pa, as the manometric fluid. The oil UIM has a full scale range of 130 Pa and is characterized by an uncertainty due to systematic effects of  $2 \text{ mPa} + 1 \times 10^{-4}$  times the pressure. The lowest absolute pressure that can be measured with this manometer is limited only by its statistical uncertainty, which is less than 1 mPa.

This development not only decreases the uncertainty of NIST primary standards at pressures below 100 Pa by as much as an order of magnitude, but also extends the range of the NIST standards downward by more than a decade in pressure. For the first time, this will enable the lowest range transducers that are critical in demanding measurement applications (e.g., in aerospace, electrical power generation, semiconductor processing, etc.) to be calibrated over their full useable range.

## ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATIONS

During the month of June, the NIST Office of Technology Innovation recommended two innovative technologies for commercialization to its Department of Energy partner under the Energy-Related Inventions Program.

A System and Method for the Ultrasonic Inspection of Tubulars and Vessels—a means of determining the presence and location of flaws in pipe structures in chemical plants, refineries, etc., based on the use of electromagnetic acoustic transducers to launch acoustic waves at ultrasonic frequencies circumferentially around the pipe.

Peripheral Mower Blade—a patented blade assembly for commercial and domestic lawn mowers, which consists of a series of square blade elements mounted

on a horizontal shaft. Each blade has four cutting edges, which are opposite to one another and provide mulching action.

## NEW STEP CLASS LIBRARY SOFTWARE AVAILABLE

Release 3.0 of the NIST STEP (Standard for the Exchange of Product data) Class Library, which implements the C++ binding of the STEP Standard Data Access Interface (SDAI, i.e., ISO 10303-23), is now complete. This software release from NIST contains many updates supporting the latest version of the emerging standard as well as support for namespacing, multischema support, enhanced handling of complex entities, and use of the STEP data with ObjectStore Object Oriented Database and Orbix (CORBA implementation).

## EMMA SUCCESSFULLY DEMONSTRATES SIMULATED NUCLEAR WASTE RETRIEVAL

The NIST easily manipulated mechanical armature (EMMA) project team has successfully demonstrated simulated nuclear waste retrieval using a prototype 10 m long, serpentine robotic arm recently developed by a private company and NIST as Cooperative Research and Development Agreement partners. The demonstration highlighted EMMA's long-reach manipulator effectively deploying a scarifying end-effector (i.e., a high pressure water jet cutter with waste retrieval suction) to retrieve hard-packed, concrete-like waste simulant and softer, more sludge-like waste simulant. The waste simulant trays were placed approximately 6 m below and at a 6 m radius from EMMA's base. During all waste retrieval passes, the EMMA manipulator performed in a very stable fashion while demonstrating its inherent dexterity and redundancy.

Development of this EMMA manipulator was sponsored by the Department of Energy's (DoE) Hanford Tank Initiative (HTI) to demonstrate innovative new technologies applied toward cleanup of underground nuclear waste storage tanks. The overall remediation plan is to deploy a 20 m EMMA manipulator from a mobile NIST RoboCrane structure over the underground tanks to allow insertion and controlled maneuverability of similar scarifying end-effectors throughout the tank volume. The RoboCrane and EMMA technologies combination is well suited to perform this task because of several shared features: they both have very favorable strength to weight ratios, they both are easily scaled for different tank sizes, and they both provide exceptional stiffness and control of payloads at very low cost.

In addition to waste retrieval applications, EMMA together with RoboCrane has other potential applications. For example, a major airplane manufacturer after seeing a demonstration of EMMA's dexterity and RoboCrane's stability, expressed an interest to apply these technologies toward their large-scale manufacturing and refurbishment processes. The EMMA team will be visiting the manufacturer's new facilities soon to further investigate potential collaboration.

### **NIST LAUNCHES STATISTICAL REFERENCE DATASETS (StRD) WEB SERVICE**

NIST announced the release of the Statistical Reference Datasets (StRD) Web site (<http://www.itl.nist.gov/div898/strd/>). This site contains reference datasets for the objective evaluation of the computational accuracy of statistical software. Users and developers of statistical software can use these datasets to ensure and improve software accuracy.

The StRD Web pages contain datasets with certified computational results in four areas: analysis of variance, linear regression, nonlinear regression, and univariate summary statistics. The collection includes both generated and "real-world" datasets, of varying levels of difficulty. Generated datasets are designed to challenge specific computations. Real-world datasets include both benign and challenging datasets from the NIST laboratories and other sources. The certification method for each statistical method is described on the corresponding Web page.

### **PHASE-LOCKED ROTATION OF ATOMIC-ION PLASMAS**

Scientists at NIST recently have phase-locked the rotation of strongly coupled nonneutral plasmas to a well-controlled rotating electric field. The plasmas, consisting of up to  $10^6$  positive beryllium ions, were contained and laser cooled in a Penning trap to near absolute zero, where the ions form a crystal that rotates as a rigid body. Bragg diffraction peaks from the rotating crystal show that the crystal lattice can remain stable for longer than 30 min or  $10^8$  rotations, and the rotation is phase locked to the rotating electric field without any slippage.

While the methods promise possible improvement for studies of plasma crystallization, the most significant practical implication is for future atomic frequency standards. The magnitude and uncertainty of the second-order Doppler shift for ions stored in a Penning trap places a severe limit on the accuracy of Penning-trap frequency standards. With this new method, the plasma can be phase locked at an optimum rotation rate

(minimum value for the mean Doppler shift), and additional measurements should allow measurement of the minimum Doppler shift to perhaps 1 %. A standard operating in this mode thus has a potential accuracy in the range of one part in  $10^{16}$  to one part in  $10^{17}$ .

### **NIST ESTABLISHES CRADA WITH PRIVATE COMPANY**

A Cooperative Research and Development Agreement has been established between NIST and a private company, to measure the performance of advanced insulation panels. Advanced insulation panels, such as compact vacuum insulation, powder or foam-filled panels, and gas-filled panels, offer the potential for thermal resistance an order of magnitude greater than current building insulation materials, such as glass-fiber. However, they have characteristics that preclude the use of traditional guarded hot plates and heat flow meters for measuring their performance. These characteristics are nonuniform surfaces and two-dimensional heat flow paths due to barrier materials used in their construction. NIST, using a laboratory equipped with a specially designed calorimeter, infrared thermography system, and a finite-element workstation, will assist the company in quantifying the performance of their prototype advanced insulation systems. The laboratory previously has been used to assist other companies in measuring the performance of advanced insulation panels.

### **PRIVATE COMPANY BEGINS COLLABORATIVE RESEARCH WITH NIST IN COMPLEX FLUIDS**

Scientists at a private company have initiated collaborative work with researchers at NIST to answer basic questions concerning the microstructure of model complex fluid systems with potential relevance to industrial applications. Two projects are under way. One focuses on model bicontinuous microemulsions based on mixtures of low and high molecular weight surfactants. Neutron scattering measurements will be used to determine characteristic domain sizes and molecular areal densities to characterize fully the nature of the microemulsion and interface. The larger goal is to improve surfactant "efficiency"—the quantity of dispersed phase microemulsified per quantity of surfactant added to the system. Maximizing surfactant efficiency, to reduce formulation costs and surfactant residue levels, would impact a variety of consumer and industrial cleaning and delivery systems.

A quite different type of aqueous suspension, consisting of dispersed clay platelets held in suspension by

electrostatic interactions with nanoscale mixed metal hydroxide particles, is the object of the other study in progress. Using NIST's in-beam couette flow scattering cell, changes in the spatial organization of the two solid constituents in the suspension is being examined in both static and kinetic conditions as a function of applied shear. The suspensions flow readily, yet form strong gels when shear stress is removed, a combination of properties of interest for enhancing the performance of drilling fluids used in horizontal drilling operations.

The key measurement strategy in both of these studies entails controlling the scattering contrast among the various phases by selectively deuterating one or more of the components. In this way the scattering from individual phases can be enhanced or suppressed to focus on particular aspects of the microstructure of these complex fluids.

#### **EUROPEAN/AMERICAN WORKSHOP ON DETERMINATION OF RELIABILITY AND VALIDATION METHODS OF NDE**

Safety is an international concern. Nondestructive evaluation (NDE) methods are used widely in in-service inspections of existing power plant, pipeline, and aircraft components; therefore, there is a need to develop quantitative methods for establishing the reliability of NDE procedures and validating such methods. NIST and the German Federal Institute for Materials Research and Testing in collaboration with the German and American Societies for Nondestructive Testing organized an international workshop on Reliability and Validation of NDE Methods. The workshop was held in Berlin on June 18–20, 1997, and was attended by nearly 100 participants from 14 countries. The agenda included invited presentations from leading European and American experts and consensus sessions. Among the subjects were: defining reliability, validation, and capability of NDE methods; essential quality characteristics of NDE; modeling of NDE processes; statistical test design; and role of human factors. A printed copy of the proceedings will be made available through the German Society for Nondestructive Testing.

#### **FEDERAL AGENCIES MEET ON LABORATORY ACCREDITATION**

The Working Group on Laboratory Accreditation (WGLA), a committee of the Interagency Committee on Standards Policy (ICSP), met at NIST Gaithersburg on June 24, 1997. The meeting was attended by representatives of 12 Federal agencies and several offices within those agencies that operate or avail themselves of laboratory accreditation programs. The ICSP

comprises all Federal agencies concerned with the development or use of normative standards and aims to ensure that the relevant provisions of the Technology Transfer and Advancement Act (TTAA) are carried out. Federal agencies have been directed to move toward more extensive use of voluntary consensus standards in lieu of developing governmental regulations or procurement standards.

The participating agencies described their accreditation activities, the standards used, and the types and numbers of accredited laboratories. The highly useful discussion led to agreement to meet periodically to consider such topics as: (1) quality system registration (ISO 9000) versus laboratory accreditation (under ISO Guide 25); (2) applicability of ISO Guide 25 to accrediting laboratories operating in accordance with Good Laboratory Practices; (3) mutual recognition agreements between U.S. and foreign accreditation bodies and their impact on trade; (4) importance of traceability to national laboratories; and (5) the role of NACLA domestically and internationally.

#### **COLABORATIVE RESEARCH PROJECT WITH ELECTROTECHNICAL LABORATORY (ETL), TSUKUBA, JAPAN**

This collaborative project involving NIST and ETL researchers is related to scanned probe microscopy (SPM) applications, specifically, scanning Maxwell-stress microscopy (SMM). A method first developed at ETL, SMM is viewed currently as the most promising candidate for integrating SPM-based fabrication methods with an appropriate in-situ electrical characterization method.

In the most recent work, the researchers investigated a set of conducting phase-separated polymer samples prepared at NIST. The idea for the project originated at NIST as a means of providing a complex, challenging materials system for evaluating specific performance and compatibility aspects of SMM.

This multidisciplinary investigation has demonstrated a novel method for visualizing how individual polymer components of each phase in the system are distributed in the quasi-two-dimensional thin-film limit. As the film thickness is reduced below about 100 nm, interfacial interactions begin to play an increasingly important role in determining the equilibrium structure of the film. Experiments on 10 nm and 100 nm thick films of pure polystyrene (ps), pure conducting polymer (poly-3-octyl thiophene, b4), and (1:9) and (2:1) blends of ps:b4, both as prepared by spin casting onto H-terminated silicon and after various annealing times and temperatures, establish the basis for the SMM contrast mechanism which permits discrimination between ps and b4 by

their different dielectric signatures in the simultaneous SMM capacitance and surface potential imaging modes. This technique can then be used to look at sub-surface mixing on the sub-micrometer scale within the 100 nm thick films as they are annealed for increasing periods of time. When combined with optical microscopy, atomic force topography, and three-dimensional imaging secondary ion mass spectrometry (SIMS), this unique information provides a significantly enhanced understanding of the complex physical and chemical interactions driving quasi-two-dimensional phase separation.

### **WEB SITE HELPS DESIGNERS OF CHILDREN'S PRODUCTS**

In a recent workshop "Systems Anthropometry" co-sponsored by NIST and the Consumer Product Safety Commission (CPSC), speakers and more than 100 attendees discussed the use and applications of human dimensional measurements, the field of anthropometry. With the support of the CPSC, NIST established a Web site called "AnthroKids," which makes available the only anthropometric survey data of children conducted in the United States. These data are valuable to designers of any type of product with which children come into contact, i.e., cars, playgrounds, cribs, etc. The NIST web site, available at <http://www.itl.nist.gov/div894/ovrt/projects/anthrokids>, provides the data in several data formats. In addition, NIST is working with several companies that make human modeling software to incorporate the data as part of their products.

### **NIST AND MULTIMEDIA COMMUNICATIONS FORUM WORK TOGETHER ON INTEROPERABILITY ISSUES OF BROADBAND COMMUNICATION TECHNOLOGIES**

On June 30, 1997, NIST hosted the meeting of the Multimedia Communications Forum (MMCF) Broadband Access Applications (BAA) Working Group. The BAA is a new working group established to identify multimedia application requirements for accessing various broadband communication technologies, including asymmetrical digital subscriber line, digital wireless, and asynchronous transfer mode. The group is focusing on a common application approach to quality of service and interoperability across a range of broadband technology offerings. NIST will be the focal point for interoperability tests for the group.

MMCF is a nonprofit research and development organization, which consists of communication service providers, applications developers, equipment vendors, and end users. The forum was founded in 1993 to

advance multimedia communications in the marketplace through a better definition of user requirements, industry interoperability demonstrations, and advancement of technical recommendations.

### **NORTH AMERICAN INTEGRATED SERVICES DIGITAL NETWORK (ISDN) USERS' FORUM (NIUF) CONSIDERS NEW ISDN APPLICATION**

At the June 1997 meeting of the NIUF, co-sponsored by NIST and ISDNworld, 115 users, implementors, and service providers of ISDN technology learned about a new work effort, Always On/Dynamic ISDN (AO/DI), and viewed demonstrations of this application. The "Always On" part allows a continual ISDN D-channel packet connection to an Internet service provider or a corporate local area network at a rate of 9.6 kbit/s. As the demand for bandwidth increases, the "Dynamic ISDN" part can use the D-channel to establish a one or two B-channel connection to allow transfer of data of rates from 64 kbit/s to 128 kbit/s, as necessary. When the bandwidth decreases, the B-channels are disconnected.

Other highlights included the approval of six documents by the NIUF, including an update to NIUF 433-94—ISDN BRI Wiring and Powering Guidelines Residence and Small Business.

### **ADVISORY BOARD RE-EXAMINES COMPUTER SECURITY ACT OF 1987**

At its June 1997 meeting, the Computer System Security and Privacy Advisory Board re-examined the Computer Security Act of 1987 and its effectiveness in improving the security of information in federal computer systems.

The Board issued three resolutions. Resolution 97-1 called for NIST to elevate its commitment to implementing the Computer Security Act of 1987 by increasing its assistance to civilian federal agencies, with emphasis on today's managerial and administrative aspects. The Board also identified five high-priority items for NIST to address on this matter. Resolution 97-2 addressed one of the five specific points in Resolution 97-1 and identified some desired attributes of a registry of security and privacy incidents.

Resolution 97-3 supports NIST's proposal to amend the Digital Signature Standard to allow for the use of additional digital signature algorithms by Federal agencies and to pursue the development of a Federal Information Processing Standard (FIPS) for public key-based key agreement and key exchange. The Board's recommendations are consistent with NIST's Federal Computer Incident Response Capability (FedCIRC) program.

**WORKSHOP ON NONNEUTRAL PLASMAS**

A NIST scientist served as a co-chairman for a four day Workshop on Non-neutral Plasmas held in Boulder July 29–Aug. 1, 1997. The workshop, with an attendance of 90 people, was sponsored by NIST, the U.S. Office of Naval Research, the National Science Foundation, and the Center for Integrated Plasma Studies at the University of Colorado.

This workshop brought together scientists with diverse interests in the trapping, cooling, and manipulation of pure ion or pure electron plasmas in electromagnetic traps. The four distinct areas discussed were (1) atomic physics studies where interests include new atomic clocks and various fundamental studies such as basic tests on trapped antimatter, (2) plasma studies that make use of the particular simplicity of nonneutral plasmas to gain information that should ultimately be applicable to other, more-complex plasma problems, (3) chemical analysis based on the concept of ion-cyclotron-resonance mass spectroscopy, and (4) non-neutral-beam studies, which have applications varying from accelerators to the generation of electromagnetic radiation.

**NIST RADIOPHARMACEUTICAL STANDARDS AID IN DEVELOPING “MAGIC BULLETS”**

NIST standards are proving crucial in a rapidly expanding area of medical research and are contributing to a new strategy of treatment for cancer, AIDS, and other diseases. The new approach employs “magic bullets,” new drugs that are biologically active molecules and designed to bind to specific cells. If these molecules can be made to carry a radioisotope, they will seek out the targeted cells and attack them, while sparing healthy cells.

Clinical trials are currently under way using a number of radioisotopes, such as  $^{90}\text{Y}$ , which has a 64 hour half-life. Antibodies labeled with  $^{90}\text{Y}$  are being investigated at several medical centers around the country as a possible treatment for non-Hodgkin's lymphoma. This form of cancer is diagnosed in about 54 000 people in the United States every year and has a 5-year survival rate of 50 % to 60 % with current treatment modalities. The results of the trials are extremely promising, although it will still be several years before these drugs will be widely available.

The existence of a NIST-traceable standard is usually required prior to FDA approval of any new radiopharmaceutical. Fortunately, radioassays of  $^{90}\text{Y}$  are based on standards and measurements already developed at NIST. These include SRM 4427B, and the NIST-determined instrument settings for commercial reentrant ionization chambers (“dose calibrators”). Other radionuclides being investigated and for which NIST has

standards available include  $^{131}\text{I}$ ,  $^{166}\text{Ho}$ , and  $^{188}\text{Re}$ . NIST plans to develop standards for two other radionuclides that can be used in such applications,  $^{64}\text{Cu}$  and  $^{177}\text{Lu}$ , over the next few years.

**NIST CONTRIBUTION RESULTS IN IMPROVED TEST STANDARD FOR CHARGING CURRENT INTERRUPTION DEVICES**

Contributions by a NIST scientist to the activities of a National Electric Vehicle Infrastructure Working Council (IWC) Personal Protection Subcommittee have resulted in an improvement to an Underwriters Laboratories standard on the testing of charging current interruption devices (CCIDs) for electric vehicle charging systems. The scientist conducted a study of how electromagnetic interference, including interference produced by the charger itself, could affect the charging current interruption device to be used in electric vehicle charging systems.

The interference issue is serious because it has the potential to terminate the charging operation at any time before the vehicle battery is fully recharged, resulting in a temporarily useless vehicle, especially in the usual overnight charging scenario. The CCID provides a safety function similar to that of the ground-fault interrupter, now mandated by the National Electrical Code for household humid environments. The IWC and its Personnel Protection Subcommittee are concerned with the development and testing of safety-related equipment for electric vehicle battery charging and requested assistance from NIST. The subcommittee was instrumental in working with Underwriters Laboratories to initiate the standard for CCIDs. The results of the scientist's study are published in NISTIR 6013, *Electromagnetic Immunity of Single-Phase Personnel Protection Devices for Electric Battery Chargers*.

**MODELING PROTEIN ELECTRON TRANSFER PATHWAYS AND DYNAMICS**

The pathways of electron transfer within and between proteins are important to the development of enzyme biocatalysts for commercial application because they determine where and how energy gets utilized in the system. Two scientists at NIST are combining computational chemistry with a mutational technique known as site-directed mutagenesis to determine probable pathways of electron transfer between protein subunits.

In the system studied (cytochrome P-450cam), electrons are transferred through three dynamically interacting protein subunits during the process of catalyzing the insertion of oxygen species into previously unreactive hydrocarbons. The computational

studies involved an extensive state-of-the-art molecular dynamics simulation of putidaredoxin (Pdx), the main cytochrome P-450cam electron transfer partner that provided a first-time dynamical picture of this protein. Multiple mutations in the gene for Pdx to change individual amino acids on the surface of the protein, along with novel enzymatic assays and the Pathways model for electron transfer, have been used to identify surface sites of interaction and probable pathways of electron transfer from the iron-sulfur center of Pdx to the heme group of P450cam. Information about solvent accessibility of various regions of Pdx and a view of multiple conformations in the C-terminal tryptophan residue are providing a molecular model that allows experimental data about Pdx-P450cam interactions to be correlated, and structure-function relationships to be tested. Further mutagenesis studies have been used to experimentally validate the computational predictions. The preliminary mutational and computational work will appear soon in the *Journal of Biological Chemistry* and in the *Biophysical Journal*, respectively.

#### **ANOTHER LOOK AT MODEL MEMBRANES**

NIST is developing new tools to take a closer look at the structure of biological membranes. New materials that mimic cell membranes are providing insight with a spatial resolution of tenths of nanometers. The cell membrane separates the inside from the outside of the biological cell, and it contains many of the functions required for life. Real cell membranes are complicated structures composed of lipids, proteins, and other molecules. Understanding membrane and membrane protein structure and function aids the development of sensors, cell surface receptor screening, biocatalysis, and other significant applications in biotechnology.

Hybrid bilayer membranes (HBMs) that are composed of both biological and nonbiological molecules are being evaluated for their suitability as mimics of cell membranes for use in studies of structure/function studies of membrane proteins. These constructs are composed of alkanethiol self-assembled monolayers and a layer of phospholipid molecules and are more rugged and versatile than other model membranes. This rugged model of a biological membrane is proving to be an excellent mimic of the real thing.

Reflection-absorption infrared spectroscopy is one of the tools used to evaluate the molecular conformation of the lipid components. In addition, neutron reflectivity provides information about the layer thickness (with a resolution of 0.1 nm to 0.2 nm), the chemical composition, and the completeness of the bilayer. By combining the use of HBMs with an improved low background,

high sensitivity neutron reflectometer, NIST researchers have been able to examine, with high resolution, single phospholipid bilayers. That they are examining single bilayers is significant since x-ray scattering from stacks of bilayer membranes has previously provided such data on model membranes. In nature, membranes do not generally occur in stacks; it is an arrangement that limits the hydration of the lipid layer. The researchers see that single bilayers in contact with bulk water are significantly more hydrated than has been determined from x-ray data. In fact, at least 10 water molecules are in contact with the hydrophilic portion of each phospholipid molecule, significantly more than the 2 water molecules to 6 water molecules indicated by measurements on multilayer systems.

#### **NIST DEVELOPS NEW CAPABILITIES FOR CHARACTERIZING ELECTROMAGNETIC PROPERTIES OF MATERIALS OVER BROADENED TEMPERATURE RANGE**

Responding to industry needs, NIST has developed a radiofrequency measurement capability to perform dielectric and magnetic characterization of bulk materials over a temperature range of 125 K to 625 K. The new capability responds to expressions of need on the part of industry for better data on materials being considered for incorporation in such electronic and electrical products as printed-wiring boards and connectors. The temperature dependence of electromagnetic parameters, especially dielectric permittivity and magnetic permeability, is a vital consideration in selecting materials for applications where significant heating or cooling can be encountered. The automobile engine compartment is one of the most challenging of these environments.

Following an initial survey of past developments relating to electrical characterization of high-frequency properties of materials at temperatures significantly above and below room temperature and of the measurement approaches in use, division staff members selected three basic methods to implement at NIST: the broadband (0.05 GHz to 8 GHz) 14 mm diameter coaxial air-line method, the coaxial re-entrant cavity method at 500 MHz and 1000 MHz, and the split-post resonator method at 2 GHz. The new capability is implemented in a system that can be used to characterize the electromagnetic properties of bulk materials, such as polymers, ceramics, and ferrites, over the specified broad temperature range. A principal component of the system is its environmental chamber, remotely programmable and controlled through an interface to a computer. To ensure that any toxic fumes produced

when specimens are heated are immediately extracted from the laboratory, the chamber is mounted beneath a laboratory exhaust hood.

### **LATEST AUTOMATED DIAMETER CALIBRATION SYSTEM AT NIST**

NIST scientists have developed a new calibration system for measuring cylinders, thread and gear measuring wires, and balls. The software system, developed by Zimmerman, allows NIST laboratory personnel to carry out a substantive measurement assurance program using an efficient and practical stand-alone personal-computer-based workstation for all reference standard master cylinders, thread and gear measuring wires, and balls. With present measurement designs, typical two standard deviation uncertainty estimates of the calibrations by mechanical intercomparison have been substantially reduced from between 85 nm and 95 nm to between 50 nm and 75 nm for steel cylinders and thread and gear measuring wires and slightly larger for balls. These figures reflect nearly a 50 % reduction over past years. Much of this reduction is a direct result of improved and frequent surveillance of a complete, yet nearly 90 % smaller, NIST calibration history.

On-line use of a new, state-of-the-art micrometer also contributes to the reduced uncertainties. The new system continues to use a special database and exclusive analysis software to record customers' measurement histories and, by that, reduce risks of reporting inaccurate measurements and improving overall measurement confidence.

### **MACHINE TOOL PERFORMANCE DATA DICTIONARY DEVELOPED**

Efficient access to relevant data on machine tools is an essential enabler of virtual machining. A NIST researcher prepared the first version of a data dictionary that contains the names and definitions of data items necessary to describe the conditions, setup, and measurement results of performance evaluation tests on machine tools. The dictionary is a repository of information describing the data to be contained in a database on machine tools. It is, therefore, metadata or "data about the data." Specifically, the data dictionary includes the names and definition of the data items in the repository, the types and sizes of these data items, and the constraints on each data item. The dictionary was developed as a database using a readily available commercial package. A user friendly interface was developed to facilitate use of the dictionary and to allow for future growth.

In its present version the dictionary contains over 250 definitions. The dictionary is being developed as part of NIST's National Advanced Manufacturing Testbed (NAMT) project on machine tool performance models and machine tool data repository. It will be tested by researchers participating in the NAMT project both at NIST and at various industrial sites. The overall goal of this project is to provide manufacturing designers and engineers with the virtual tools required to predict machine tool performance and parts accuracy with minimal prototyping.

### **COLLABORATIVE RESEARCH ON CUBANE**

A NIST/University of Chicago collaboration is studying the structure and dynamics of solid cubane using x-ray and inelastic neutron scattering techniques at the NIST Center for Neutron Research. There are potential explosive and pharmaceutical applications of cubane and its derivatives, progress on which has slowed to a standstill due to lack of funds and basic research. This makes the recent results of the NIST/University of Chicago collaboration, which were published in the June 30 issue of *Physical Review Letters* Vol. 78, pp. 4938-4941 (1997), both timely and important. In this study, the researchers were able to measure the high temperature structure of solid cubane and then successfully model the intermolecular interactions using a phenomenological potential energy function that correctly predicted both the low and high temperature crystal structures of cubane.

### **IN SITU CHARACTERIZATION OF CORROSION AT ELEVATED TEMPERATURES AND PRESSURES**

Researchers at NIST recently have demonstrated the use of in situ Raman spectroscopy to characterize corrosion of metals in supercritical water, where optical access is extremely challenging. Metal corrosion at elevated temperatures (600 K to 900 K) and pressures (17 MPa to 50 MPa) is very important in systems such as supercritical water power plants, nuclear power plants, and hydrothermal oxidation reactors. Since significant differences can exist between the corrosion species that occur at elevated temperatures and pressures compared to those observed "dry" or under ambient conditions, in situ measurements are needed to understand the corrosion mechanisms in these systems. However, due to the hostility of the environment at supercritical conditions, most corrosion monitoring techniques are not applicable to in situ investigations.

A specially designed optical flow cell was constructed that allows the measurement of Raman spectra



of surfaces in a flowing solution. The cell utilizes off-the-shelf high temperature/pressure “fittings” and provides three optical ports with a unique, NIST developed, gold seal for the optical windows. The window surfaces exposed to the solution are chemical vapor deposited diamond films to resist corrosion. In situ Raman spectra were collected from a number of metals in water at pressures of 25 MPa and at a series of temperatures ranging from room temperature to 800 K, i.e., conditions approximating a hydrothermal oxidation reactor. A variety of corrosion species were identified at the high pressure/temperature conditions that were not observable at ambient conditions or in post exposure ex situ tests (which also utilized techniques such as x-ray diffraction, scanning electron microscopy, or energy dispersive x-ray spectroscopy). The results found from the in situ measurements are in general agreement with accepted corrosion mechanisms. Future studies will involve nickel-base and zirconium-base alloys at temperatures and pressures selected to simulate conditions encountered in power generation systems.

#### **ELECTRON-IMPACT IONIZATION CROSS SECTIONS NOW AVAILABLE ONLINE**

A World Wide Web (WWW) database of electron-impact ionization cross sections has been developed at NIST and is now available online. These data are needed in a wide range of applications, such as modeling plasmas used in the processing of semiconductors, designing mercury-free fluorescent lamps, assessing the efficiency of ion gauges, normalizing mass spectrometer output, diagnosing plasmas in magnetic fusion devices, and modeling radiation effects on materials.

The database derives from a series of articles by NIST scientists and collaborators. They apply the Binary-Encounter-Bethe (BEB) model, which is specifically designed for electron-impact ionization, and which is versatile enough to provide cross sections for atoms as well as molecules. The theory does not use any fitting parameters and provides a simple analytic formula for the ionization cross section per atomic/molecular orbital. The database provides the analytic formula and a means of calculating the cross section at a given energy. The cross sections are presented both as graphical plots and in tabular form. Initially, data are provided for two atoms and 39 industrially important molecules. More atoms, molecules, and ions will be added later.

The electron-impact ionization database may be accessed on the WWW at <http://physics.nist.gov/PhysRefData/Ionization/Xsection.html>. The complete set of databases provided by NIST may be accessed at <http://physics.nist.gov/PhysRefData/contents.html>.

#### **NEW MEASUREMENT SERVICE IMPROVES AIRCRAFT SAFETY**

Researchers at NIST have introduced a new measurement service to address an urgent need in aviation safety. Aircraft are equipped with flashing “anticollision” lights to provide a visual warning to other pilots of an aircraft’s location. However, a lack of visibility of some such lights has been cited by the National Transportation Safety Board as a contributing factor in airliner crashes, including the a crash at Los Angeles International Airport (LAX) in 1991. The problem has received wide media attention, including an investigative report by a Television News Program last summer.

To help solve the problem, NIST worked with the Federal Aviation Administration, which specifies the requirements for the effective intensities of the anticollision lights and enforces the maintenance of these lights on all commercial aircraft. Prior to NIST’s involvement, there had been large variations in measurement results due to the absence of standardized measurement procedures and calibration standards. Now there are established flashing-light photometric standards and a new measurement service for calibrating photometers for both white and red anticollision lights.

The flashing-light photometric unit (lx.s) has been realized based on the NIST detector-based candela. The relative expanded uncertainty of the NIST flashing-light standards in this measurement is 0.6 %. This work was reported recently to the aviation industry at a scientific conference.

#### **NIST DEVELOPS VIRTUAL REALITY MODELING LANGUAGE (VRML) PARSER AND SCENE GRAPH GENERATOR**

To determine the effectiveness of automated test generation methods to develop conformance tests for specifications of standards for computer software, NIST has developed Viper, a Java-based VRML parser and scene graph generator, that provides both syntax-and semantic-level checking of VRML files. VRML is the file format standard for three-dimensional multimedia and shared graphics (i.e., virtual worlds) on the Internet. Viper uses the JavaCC parser generator tool, a Java counterpart to the popular Unix lex/yacc tools. Both source and binary distributions of this first release of Viper are available from: <http://autumn.ncsl.nist.gov/vts/Viper/readme.html>

Viper includes a comprehensive set of capabilities and features for validating VRML files. In addition to performing complete syntax checking, Viper supports scene graph construction as well as validating semantic correctness, such as hierarchical checking for all VRML

nodes, DEF/USE and ROUTE statement checking, and range of value checking. Unique to Viper, the scene graph construction permits a user to step through the parse tree, thereby providing the ability to view and eventually (in future releases) interactively debug a VRML file.

As part of the VRML conformance testing project, Viper will be used to validate the correctness of the VRML Test System (VTS) tests. In the next year, Viper will be used as an automated test generation tool, in producing VTS tests.

### **NIST ESTABLISHES ONLINE CONFORMANCE TESTING DIRECTORY**

NIST has established a Web-based Directory of Conformance Testing Programs, Products, and Services, which provides sources for testing software for conformance to information technology (IT) standards. It includes conformance testing information for a variety of IT standards, including Federal, national, and international standards.

The online directory provides a central information source for linking providers of conformance testing materials with the users of these materials. The publication contains data sheets from suppliers describing the conformance testing materials that they offer. The directory provides tables for easily cross-linking IT standards with available conformance testing program sponsors, test suites, certificates, and testing services. The directory can be used to locate: conformance testing/certification programs; test services offered in both the public and private sectors; listings of products that have been tested; and various test suites and tools that are used to measure conformance to IT standards.

The directory currently contains conformance testing information for IT standards in the areas of computer security, database languages, operating systems, programming languages, and computer graphics. The Web site is <http://www.nist.gov/ctdirectory.html>.

### **VALIDATION PROCEDURE DEVELOPED FOR COMPACT THERMAL MICROELECTRONIC PACKAGE MODELS**

NIST scientists have developed the first validation procedure for compact models for system simulations of natural-cooled systems. This is important for handheld and portable equipment, such as laptop computers and cellular and wireless telephones. Computer-aided thermal design is becoming more important for electronic systems. To ensure timely, efficient simulations, the thermal details of the microelectronic packages must be minimized by using compact (as opposed to

detailed) thermal models. These models take on various forms, including simple block-like structures and thermal resistance networks. For compact models to be used widely, methods are needed to assess the accuracy and applicability of the models when applied to different operational environments, for example, connected or not connected to a heat sink, natural or forced convection, vertical or horizontal mounting.

The NIST team used a standard enclosure for natural convection experiments with single packages to develop the procedure, together with both detailed and compact model simulations, temperature measurements using special packaged thermal test chips and thermocouples, and flow visualization. They demonstrated using a 3×3 array of thermally interactive packages in a narrow aspect-ratio enclosure that package models validated in the standard enclosure could give accurate thermal results in a realistic application. The enclosure approximated the dimensions of a laptop computer, about 18 cm×18 cm×2.5 cm. NIST anticipates that libraries of compact models will be implemented as part of commercial thermal simulators and that the NIST validation procedure will be used in their development.

### **WIRE BONDING “BIBLE” REVISED AND EXPANDED**

A NIST scientist has authored a greatly revised and updated edition of his 1989 wire bonding handbook. The new work, *Wire Bonding in Microelectronic—Materials, Processes, Reliability, and Yield*, has just been published by a commercial publisher as giving the “practical know-how you need to design and evaluate wire bonds engineered with the latest—and still evolving—metallurgies,” the book provides step-by-step instructions on how to exploit new higher density interconnection techniques and engineer reliable bonds at a very high yield. For example, hands-on guidance is provided to test wire bonds, to clean bond pads to improve bondability and reliability, and to solve cratering, heel cracks, bond fatigue, so-called purple plague, and other mechanical problems. The work provides contemporary details on utilizing fine-pitch bonding, applying new bonding metallurgies, and exploring wire sweep and the wire bonding mechanism.

### **WORKSHOP ON CHARACTERIZATION AND MODELING OF POLYMER/POLYMER INTERPHASE**

A workshop on “Characterization and Modeling of Polymer/Polymer Interphase” was held at NIST June 12, 1997. The purpose of the workshop was to identify industry needs relating to characterization and modeling

of the polymer/polymer interphase regions and to draft a research agenda for meeting industry needs. The 40 participants included 17 from industry and six from academia.

Following the presentations on fundamental issues and industrial perspectives on characterization and modeling of the interface/interphase in polymer/polymer systems, three working groups were formed to discuss: (1) coating systems, (2) service life/durability, and (3) processing. Research needs identified in the working group sessions were reported at the final plenary session and will provide the basis for a research agenda addressing industry needs, which will be published in the workshop report. It was proposed that a follow-up meeting be held to discuss how the research agenda should be implemented.

## **SECOND INTERNATIONAL CONFERENCE ON FIRE RESEARCH AND ENGINEERING**

NIST joined with the Society of Fire Protection Engineers to organize the Second International Conference on Fire Research and Engineering (ICFRE2). The conference, which was held at NIST in August 1997, serves as a forum for the exchange of information among leaders in fire research and practitioners in the field of fire safety engineering. More than 220 people participated in the conference, including representatives from fire research organizations across the United States and from Australia, Belgium, Canada, Denmark, Finland, France, Great Britain, Greece, Hong Kong ROC, New Zealand, Sweden, and Taiwan ROC. Papers were presented on fire modeling, fire growth, performance-based fire safety engineering, fire suppression, smoke management, fire detection, and firefighter safety. Given the great success of this conference, NIST and the Society of Fire Protection Engineers are planning ICFRE3, which will be held in Chicago during the week of Aug. 16, 1999.

## **STANDING ROOM ONLY AT NCSL FOR PAPER ON UNCERTAINTIES**

A paper presented at the 1997 National Conference of Standards Laboratories' Annual Meeting held in Atlanta, GA, in July 1997 by a NIST scientist and entitled "The Guidelines for Expressing Measurement Uncertainties and the 4:1 Test Uncertainty Ratio (TUR)," attracted considerable attention. Its subject was the transition in the methods for calculating uncertainties. In 1988, MIL-STD-45662A adopted the 4:1 TUR; this was later (1994) incorporated into the ANSI/NCSL Z540-1 standard. However, in 1992, NIST began to adopt a new method to evaluate and express measure-

ment uncertainties. This method uses the root-sum-of-squares (RSS) method for calculating the combined standard uncertainty from the Type A and Type B uncertainty components instead of simple arithmetic addition, and a coverage factor  $k = 2$  for calculating the expanded uncertainty (equivalent to two standard deviations) instead of the three standard deviations often used in the United States.

The change from the arithmetic addition to the RSS method has reduced significantly the calculated standard uncertainties. The change of the coverage factor from  $k = 3$  to  $k = 2$  further reduces the calculated expanded uncertainties by 33 %. In many cases, the combined effects have caused a 50 % or more reduction of calculated uncertainty from the same uncertainty budget. This has been documented in calibration and measurement reports. The reduced uncertainties also have caused significant increases of the actual TURs for the same measurement processes and tolerance ranges. For many industrial institutions, the decreased uncertainty and increased actual TUR represent an improvement of our knowledge of the measurement uncertainties, rather than an improvement in measurement quality. Therefore, when establishing the TUR for measurement quality control, the method used for calculating measurement uncertainty is an important consideration. In the paper, some examples are used to demonstrate the results of using different uncertainty methods for calculating measurement uncertainty and TUR from the same uncertainty budget.

## **NIST TO PROVIDE TRACEABILITY LINKAGE AND OVERSIGHT OF PRIVATE SECTOR/ STATE PROVISION OF PROFICIENCY TEST SERVICES FOR EPA/STATES WATER PROGRAMS**

Since the 1970s, the Environmental Protection Agency (EPA) has conducted proficiency testing of public and private-sector laboratories that conduct analyses for the various water programs administered by the states and EPA under the Clean Water and the Safe Drinking Water Acts. The results of this testing are used by the EPA and states as part of their assessment of a laboratory's competence to conduct analyses and to produce meaningful and reliable environmental data. At the end of 1998, plans are to move from the current program in which these water proficiency testing studies are conducted by EPA itself to a program in which interested states and private companies would provide these proficiency testing services with appropriate government oversight.

In a new government-private sector partnership, NIST is working with EPA, states, and other public and

commercial entities to establish a system under which private sector and interested states can be accredited by NIST to provide NIST-traceable proficiency testing to those laboratories testing drinking water and wastewater for regulated chemical, microbial, radiological, and toxicological parameters. As part of this joint effort to externalize and improve the nation's environmental laboratory proficiency testing programs, EPA and the states will specify the requirements of the water proficiency testing studies, including the acceptability criteria for environmental testing laboratory performance; private-sector and state providers will develop and manufacture proficiency testing materials, and conduct the proficiency testing studies of the environmental testing laboratories; and NIST will establish a program to provide NVLAP accreditation to those commercial/state providers that can demonstrate their capabilities and competence to supply water proficiency testing studies that meet the needs of EPA and the states. In addition, NIST will provide Standard Reference Materials to assist suppliers in value-assigning their proficiency testing samples along with sample audits to establish NIST traceability of these measurements.

To avoid a lapse in the availability of appropriate proficiency testing studies, NIST personnel are working to set up the infrastructure in time for the first class of private-sector and state water proficiency testing providers to become NIST accredited by January 1999.

#### **ANSI/OAS WORKSHOP-AUTOMATION IN STANDARDS ORGANIZATIONS**

A NIST representative participated in a workshop sponsored by the American National Standards Institute (ANSI) and the Organization of American States (OAS) in July 1997, in New York City. The workshop, on the need for automation in a standards organization, provided participants with overviews of current information technology activities, specifically related to the Internet and World Wide Web, in ANSI, ISO, and IEC. The NIST representative discussed standards information and inquiry requirements and responsibilities under the WTO Agreement on Technical Barriers to Trade and ISO Information Network (ISONET) as well as how automation can assist countries in enhancing and fulfilling these responsibilities.

ANSI, ISO, and IEC representatives demonstrated their homepages to illustrate the use of the web in providing users with catalog information, standards under development, technical committee contacts, and other standards-related information.

Participating OAS countries were Argentina, Bahamas, Bolivia, Brazil, Canada, Chile, Colombia, Costa Rica, Ecuador, El Salvador, Grenada, Guatemala, Guyana, Honduras, Jamaica, Mexico, Panama, Paraguay, Peru, Dominican Republic, St. Lucia, Trinidad and Tobago, United States, Uruguay, and Venezuela.

#### **NIST HOSTS WORKSHOP ON DIGITAL LIBRARY OF MATHEMATICAL FUNCTIONS**

NIST hosted an invitational workshop in Gaithersburg in July 1997, to develop plans for the NIST Digital Library of Mathematical Functions (DLMF). The DLMF is envisioned as a modern replacement for the NBS Handbook of Mathematical Functions, which was first issued in 1964. More than 150 000 copies of the handbook have been sold by the U.S. Government Printing Office and several commercial publishers (it remains available today from these sources).

The handbook contains technical information, such as formulas, graphs, and tables, on a variety of mathematical functions of widespread use in the sciences and engineering. The new DLMF would revise and expand this core data and make use of advanced communications and computational resources to disseminate the information in ways not possible using static print media. Examples include formulas downloadable into symbolic systems, dynamic graphics, reference algorithms and software, tables generated on demand, and application modules tailored to specific domains such as quantum physics.

Workshop participants included a number of well-known experts in special functions and their application.

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### **Standard Reference Materials**

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#### **SRMs IMPROVE SILICON WAFER RESISTIVITY TESTING**

The resistivity of silicon wafers is a vital concern to the multibillion dollar semiconductor industry. NIST is releasing five new Standard Reference Materials that will enable manufacturers to calibrate resistivity test instruments to 0.3 % or better, with 95 % confidence. This advance, an improvement over previous resistivity SRMs, cuts resistivity measurement uncertainty by approximately a factor of five. The upgraded measurement capacity significantly surpasses the accuracy and precision requirements set forth at the 1991 SEMATECH Workshop on Silicon Materials for Mega-IC Applications.

The new standards will have better uniformity of resistivity and thickness, larger characterized area and smaller certification uncertainty than previous resistivity standards. The soon-to-be-distributed SRMs are 100 mm in diameter and are intended for the calibration or performance verification of four-point probes and eddy current testers. Unlike the current resistivity SRMs that are certified for value only at the wafer center, the new SRMs provide certified measurements at the center and on circles of 10 mm and 20 mm diameters for better compatibility with automated resistivity uniformity mapping instruments.

The standards are: SRM 2541 at 0.01  $\Omega$  cm, SRM 2542 at 0.1  $\Omega$  cm, SRM 2445 at 25  $\Omega$  cm, SRM 2546 at 100  $\Omega$  cm, and SRM 2547 at 200  $\Omega$  cm.

For more information, contact James Ehrstein, B310 Technology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2060, <j.ehrstein@nist.gov>. To purchase the SRMs when available, contact the Standard Reference Materials Program, Building 202, Rm. 204, NIST, Gaithersburg, MD 20899-0001, (301) 975-6776, fax: (301) 948-3730, <srminfo@enh.nist.gov>.

#### **STABLE RETARDER IS NEWEST SRM FOR OPTICS INDUSTRY**

Optical retarders, known as waveplates, are critical components for polarization control in the optics industry. They play an important role in optical sensor, data storage, medical and military systems as well as a variety of test and measurement instruments. Waveplate manufacturers typically measure retardance using laboratory benchtop systems with varying uncertainty. Customers also require different levels of retarder uncertainty—ranging from greater than 10 % for multiple-order waveplates to less than 0.1 % for critical devices.

At the urging of several manufacturers, scientists at NIST developed and demonstrated a stable linear retarder for use as a calibration reference. The device is a nominally quarter-wave retarder at 1.3  $\mu\text{m}$ ; this wavelength was chosen to meet fiber optic telecommunication needs. The retarder's phase shift is stable within 0.1° over a wavelength range greater than 50 nm, an input angle range greater than 1°, and a variation in room temperature greater than 10 °C.

The devices are packaged in a protective housing that ensures retardance stability in the presence of humidity and contamination. NIST estimates the retardance will remain stable within 0.01° for 10 years when the device is stored properly in typical laboratory conditions. Each device will be individually measured and a certified retardance value with an expanded uncertainty less than 0.08° reported.

The retarder is offered for \$5342 as Standard Reference Material 2525. It is available from the Standard Reference Materials Program, Building 202, Room 204, NIST, Gaithersburg, MD 20899-00001, (301) 975-6776, <srminfo@nist.gov>.

#### **REFERENCE MATERIAL BEING DEVELOPED FOR p53 MUTATION DETECTION IN HUMAN CANCER**

NIST scientists are currently developing a Standard Reference Material (SRM) for the diagnosis of p53 mutations. Interest in the prototype p53 reference material already has been expressed by many leading companies who have developed mutation detection technologies. The p53 gene is mutated in human cancer in more than half of all tumors. Additionally, research in leading cancer laboratories indicates that p53 mutation can serve as a prognostic indicator for breast cancer. Long-term survival in breast cancer is more likely in patients with a wild type (mutation negative) p53 status in their tumors than those having a mutant p53 gene. This correlation may extend to other human cancers as well. The American Cancer Association predicted that over 1.6 million new cancers would be detected in 1996 and even more in 1997. At least 800 000 of those are likely to contain mutations within p53.

Inexpensive, definitive tests for mutations within this gene are currently unavailable, and large-scale diagnostic testing for mutations is unlikely to occur until the diagnostic industry develops such tests. Thus, there is a strong demand for a defined reference material to help test the efficacy of the tests. The reference material being developed at NIST will contain a panel of clones, each containing a different mutation. The clones to be included in the SRM panel are being selected from a large number of clones created at NIST by a technique known as site-directed mutagenesis. The final configuration of the SRM panel will include clones containing mutations most commonly found in human cancer, as well as others that are difficult to detect by some of the currently available mutation detection assays. The results of a collaborative study between NIST and a private company comparing two such mutation detection technologies was presented recently at the 4th International Workshop on Mutation Detection, Czech Republic. The use of the SRM should expedite the development of diagnostic tests for p53, and high-quality measurements are anticipated from its availability.

# Calendar

March 23–27, 1998

## CHARACTERIZATION AND METROLOGY FOR ULSI TECHNOLOGY

**Location:** National Institute of  
Standards and Technology  
Gaithersburg, MD

**Sponsors:** NIST, SEMATECH, Semiconductor Research Corporation, and American Vacuum Society.

**Audience:** Semiconductor research and development staff in the electronics industry.

**Format:** Invited oral presentations, contributed poster papers, panels, and equipment exhibition.

**Purpose:** To bring together scientists and engineers interested in all aspects of the technology and characterization techniques for semiconductor device research, development, manufacturing, and diagnostics: chemical and physical, electrical, optical, in-situ, and real-time control and monitoring.

**Topics:** The workshop will provide a forum to present and discuss critical issues, problems, evolving requirements and analysis needs, future directions and key measurement principles, capabilities, applications, and limitations.

**Technical Contact:** David Seiler, NIST, Building 225, Room B344, Gaithersburg, MD 20899-0001, phone: 301/975-2054, fax: 301/948-4081, email: david.seiler@nist.gov, WWW Homepage: <http://www.eeel.nist.gov/812/dgs.html>.

May 4–5, 1997

## METROLOGY FOR ELECTRONIC PACKAGING AND INTERCONNECTION

**Location:** National Institute of  
Standards and Technology  
Gaithersburg, MD

**Sponsor:** NIST.

**Audience:** Industry, university, and government experts in electronic packaging and interconnection.

**Format:** Technical presentations and working groups.

**Purpose:** To revise NIST's strategic planning in electronic packaging and interconnection based upon 1997 editors of leading industrial roadmaps.

**Topics:** Electronic packaging, metrology, interconnection, printed wiring boards, on-chip interconnects, semiconductor packaging, and materials.

**Technical Contact:** Michael Schen, NIST, Building 224, Room B320, Gaithersburg, MD 20899-0001, phone: 301/975-6741, fax: 301/869-3239, email: michael.schen@nist.gov.

July 5–10, 1997

## 1998 CONFERENCE ON PRECISION ELECTROMAGNETIC MEASUREMENTS

**Location:** Washington Renaissance Hotel  
Washington, DC

**Sponsors:** NIST, Bureau International des Poids et Mesures, IEEE Instrumentation and Measurement Society, National Research Council of Canada, and Union Radio Scientifique Internationale.

**Audience:** Electromagnetic metrologists, especially from national laboratories; physicists in the fundamental constants area; and instrumentation developers.

**Format:** Verbal paper sessions, poster paper sessions, and ad hoc working group meetings.

**Purpose:** To provide a forum in which the international metrology community can discuss the most recent developments in physics and electrotechnology that apply to precision electromagnetic measurements and international standardization.

**Topics:** Antennas and dielectrics, automated measurement systems, DC/LF measurements and standards, fundamental constants, international compatibility of measurements, microwave/mmwave measurements (RF and EMC), nanotechnology (cryoelectronics), optical wavelength metrology, power and energy, quantum standards, realizations of units, and time and frequency.

**Technical Contact:** Norman Belecki, NIST, Building 220, Room B146, Gaithersburg, MD 20899-0001, phone: 301/975-4223, fax: 301/926-3972, email: norman.belecki@nist.gov, WWW Homepage: <http://www.eeel.nist.gov/cpem98>.

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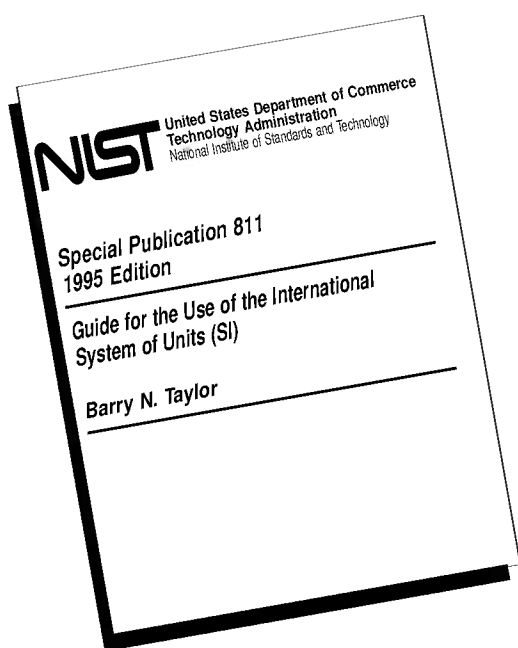
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# The International System of Units (SI)

## *The Modern Metric System*



Uncertain about the International System of Units (universally abbreviated SI), the modern metric system used throughout the world? Do you need to know the proper way to express the results of measurements and the values of quantities in units of the SI? Do you need to know the NIST policy on the use of the SI? Then you need the 1995 edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*.

The 1995 edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*, by Barry N. Taylor, is now available.

The 1995 edition of SP 811 corrects a number of misprints in the 1991 edition, incorporates a significant amount of additional material intended to answer frequently asked questions concerning the SI and SI usage, and updates the bibliography. The added material includes a check list for reviewing the consistency of written documents with the SI. Some changes in format have also been made in an attempt to improve the ease of use of SP 811.

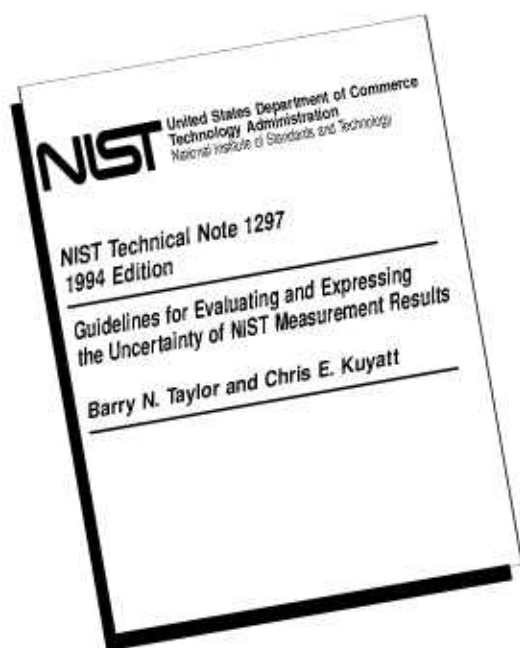
The topics covered by SP 811 include:

- NIST policy on the use of the SI in NIST publications.
- Classes of SI units, those SI derived units that have special names and symbols, and the SI prefixes that are used to form decimal multiples and submultiples of SI units.
- Those units outside the SI that may be used with the SI and those that may not.
- Rules and style conventions for printing and using quantity symbols, unit symbols, and prefix symbols, and for spelling unit names.
- Rules and style conventions for expressing the results of measurements and the values of quantities.
- Definitions of the SI base units.
- Conversion factors for converting values of quantities expressed in units that are mainly unacceptable for use with the SI to values expressed mainly in units of the SI.
- Rounding numbers and rounding converted numerical values of quantities.

Single copies of the 84-page SP 811 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.



# Evaluating and Expressing the Uncertainty of Measurement Results



Uncertain about expressing measurement uncertainty? Do you need to know how NIST states the uncertainty of its measurement results and how you can implement their internationally accepted method in your own laboratory? Then you need the newly available 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*.

The 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, by Barry N. Taylor and Chris E. Kuyatt is now available.

The 1994 edition of TN 1297 includes a new appendix—Appendix D—which clarifies and gives additional guidance on a number of topics related to measurement uncertainty, including the use of certain terms such as accuracy and precision. Very minor word changes have also been made in a few portions of the text of the 1993 edition in order to recognize the official publication in October 1993 by the International Organization for Standardization (ISO) of the *Guide to the Expression of Uncertainty in Measurement* on which TN 1297 is based. However, the NIST policy on measurement uncertainty, Statements of Uncertainty Associated with Measurement Results, which is reproduced as Appendix C of TN 1297, is unchanged.

It is expected that the 1994 edition of TN 1297 will be even more useful than its immediate predecessor, the 1993 edition, of which 10 000 copies were distributed worldwide.

Those United States readers who wish to delve into the subject of measurement uncertainty in greater depth may purchase a copy of the 100-page ISO *Guide* from the Sales Department of the American National Standards Institute (ANSI), 105-111 South State Street, Hackensack, NJ 07601. Copies may also be purchased from the ISO Central Secretariat, 1 rue de Varembé, Case postale 56, CH-1211 Genève 20, Switzerland.

Single copies of the 20-page TN 1297 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.

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